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SOME NEGLECTED PHENOMENA IN THE HEAT TREATMENT OF STEEL.

By M. E. Leeds.

SUMMARY.

Steel specimens ranging in size from 12-in. round to 2-in. round were brought to four furnace temperatures, 1000, 1200, 1400 and 1600° F. Continuous records were kept showing the rates of heating of the various specimens and the relation in temperature between the furnace gases, the surface of the specimen, and various points in its interior. It was found that the specimens assumed the furnace temperature in less time as the temperatures increased (except when the furnace temperature was slightly above the transformation point); that the temperatures throughout the interior of the specimens equalized themselves rapidly; that a contact thermocouple may be used to show when the specimen has reached the furnace temperature throughout its mass; and that a contact couple may be used to show when the specimen passes through its transformation point.

SOME NEGLECTED PHENOMENA IN THE HEAT TREATMENT OF STEEL.

By M. E. Leeds.

The experiments described in this paper were undertaken to determine the variations in rates of heating of specimens of different sizes to various furnace temperatures, and particularly to determine the relation in temperature between the atmosphere

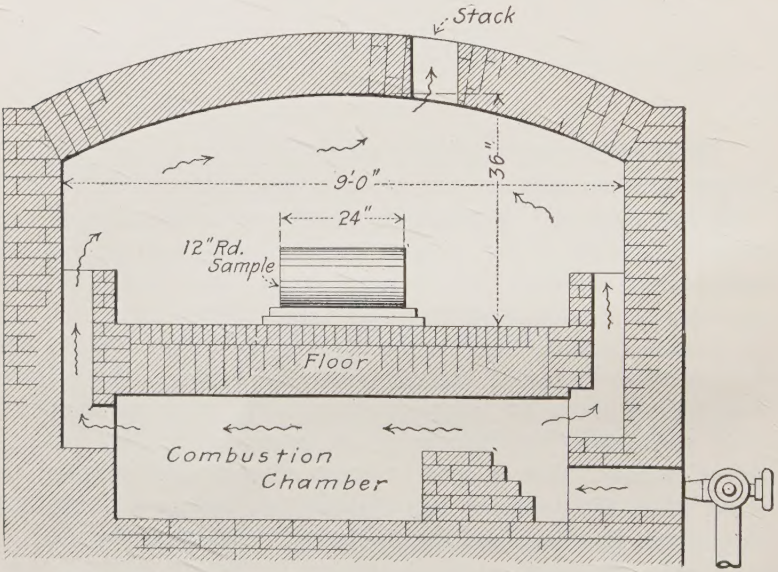


Fig. 1.—Diagrammatic Section of Furnace, Showing Location of Specimen.

of the furnace, the surface of the specimen and various points in the interior of the specimen. The phenomena discussed are referred to as neglected phenomena, because it has seemed to the writer that many of those engaged in the heat treatment of steel, who have been at much pains to investigate physical properties, to determine critical temperatures and to find out on small samples in the laboratory the most favorable tempera-

tures at which to treat, have neglected a study of the furnace conditions, and the phenomena of heat transfer to the specimen and of heat flow in it. A thorough understanding of these phenomena is quite essential to a precise control of the temperature of the specimen and must be understood in order to bring it up to the desired treating temperature at a fairly uniform rate, and to determine from measuring instruments when this temperature has been attained. Thermocouple pyrometers, which are the instruments almost universally used, can only show the temperatures of their hot ends, and it is very important to know

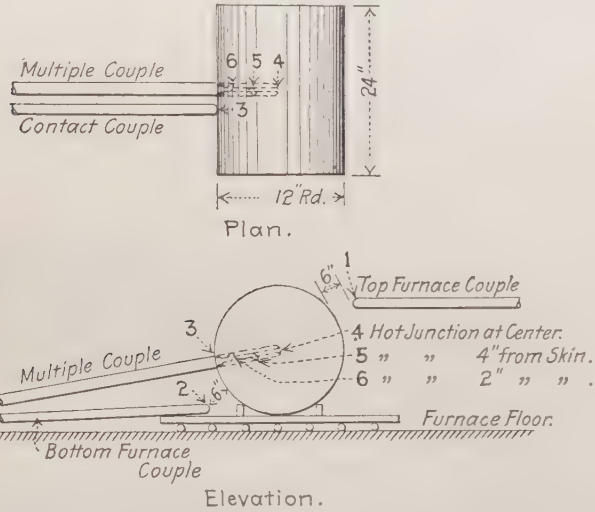


FIG. 2.—Thermocouple Arrangement for Specimen 12 in. in Diameter.

under what conditions the temperatures of the interior of the specimen may be correctly inferred from the pyrometer reading.

The experiments cover round specimens of normal open-hearth carbon steel (0.5 per cent carbon), ranging in size from 12 in. diameter by 24 in. long, to 2 in. diameter by 24 in. long. Each specimen was heated to four temperatures, namely, 1000, 1200, 1400 and 1600° F., and during the time of heating a continuous record was kept of the furnace temperatures, the temperature of the surface of the specimen, and of one to three points in its interior. The interior points were 2, 4 and 6 in. in from the surface.

These experiments were made at the plant of the Midvale Steel Co. in the summer and fall of 1914.

METHOD OF THE EXPERIMENTS.

The temperature of the furnace, controlled by two pyrometers, was first brought to that to which the specimen was to be heated, and held there at least one-half hour. The cold specimen was then placed in the furnace. This immediately lowered the furnace temperature, and it was again brought up as quickly as possible to that desired and held there until the

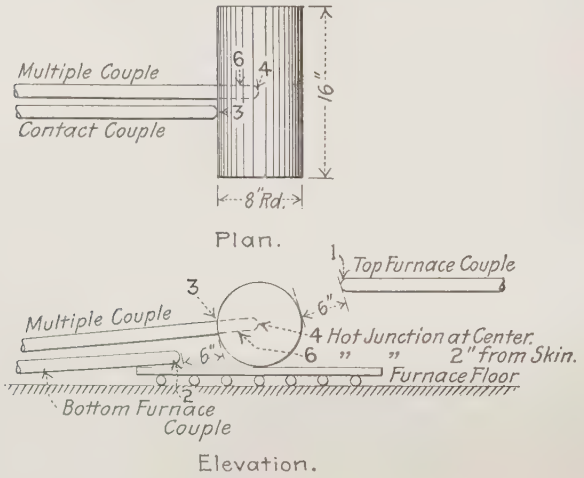


FIG. 3.—Thermocouple Arrangement for Specimen 8 in. in Diameter.

specimen assumed approximately the same temperature. Four specimens were used in the experiments. Three of them were brought to four different temperatures, and the 8-in. specimen was brought to six different temperatures. There are here presented the results of 18 runs.

During each run on the 12-in. specimen, continuous temperature records were taken of six positions, two in the furnace, one at the surface of the specimen and three in the interior. On the 8-in. specimen continuous records of five different positions were taken, and on the 4-in. and 2-in. specimens, four continuous records were taken on each.

A Rockwell oil-burning furnace was used. Its construction and the way in which the test specimens were placed is indicated in Fig. 1. The disposition of the thermocouples for each experiment is shown in Figs. 2, 3 and 4.

The furnace temperatures and the rates of heating of the various parts of the different specimens are shown in Figs. 5 to 9, inclusive.

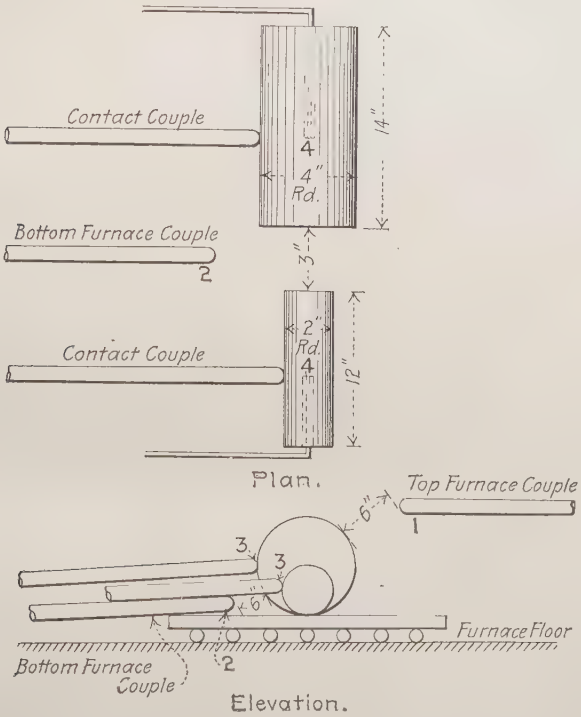
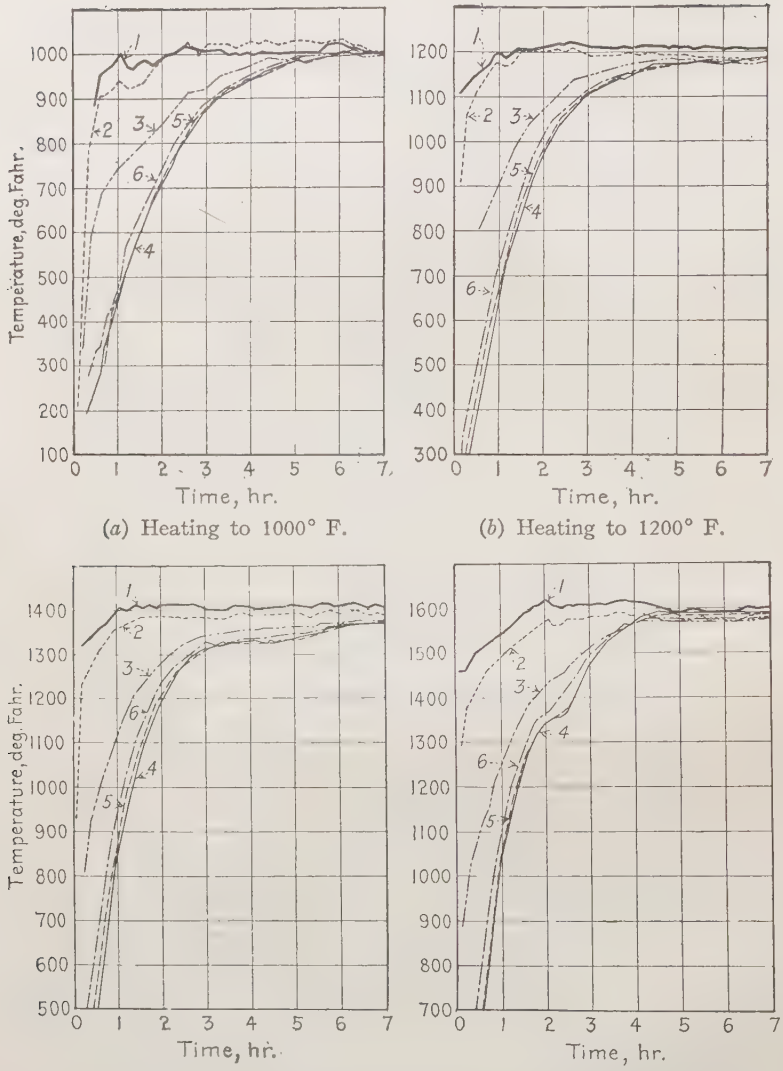


FIG. 4.—Thermocouple Arrangement for Specimens 4 and 2 in. in Diameter.

Instruments—The temperature records were taken on a multiple-point printing recorder of the potentiometer type. The thermocouples were iron-constantan, and both the couples and the potentiometer were frequently checked for accuracy during the progress of the experiments. The device used for taking the interior temperatures was a multiple couple made with a single iron wire, to which were attached at 2-in. intervals



(*a*) Heating to 1000° F.

(*b*) Heating to 1200° F.

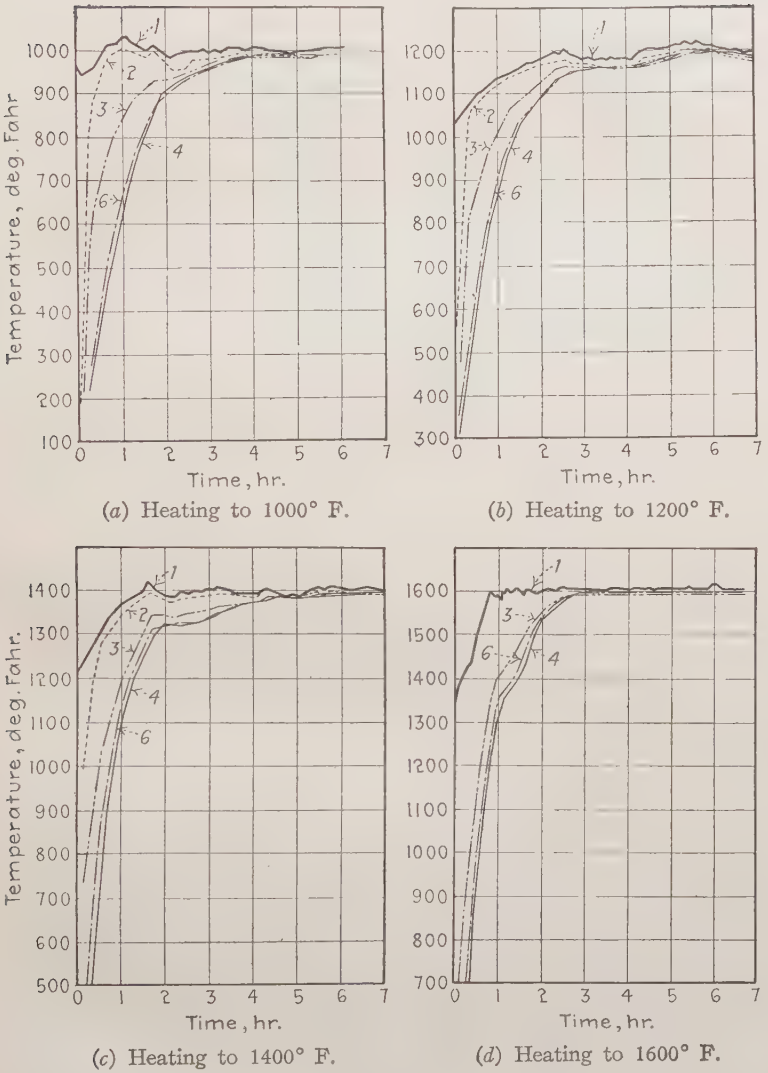
(*c*) Heating to 1400° F.

(*d*) Heating to 1600° F.

LEGEND.

1....Upper furnace couple.
2....Lower furnace couple.
3....Contact couple.
4....Couple at center.
5....Couple 4 in. from surface.
6....Couple 2 in. from surface.

FIG. 5.—Rates of Heating 12-in. Specimen.



(*a*) Heating to 1000° F.

(*b*) Heating to 1200° F.

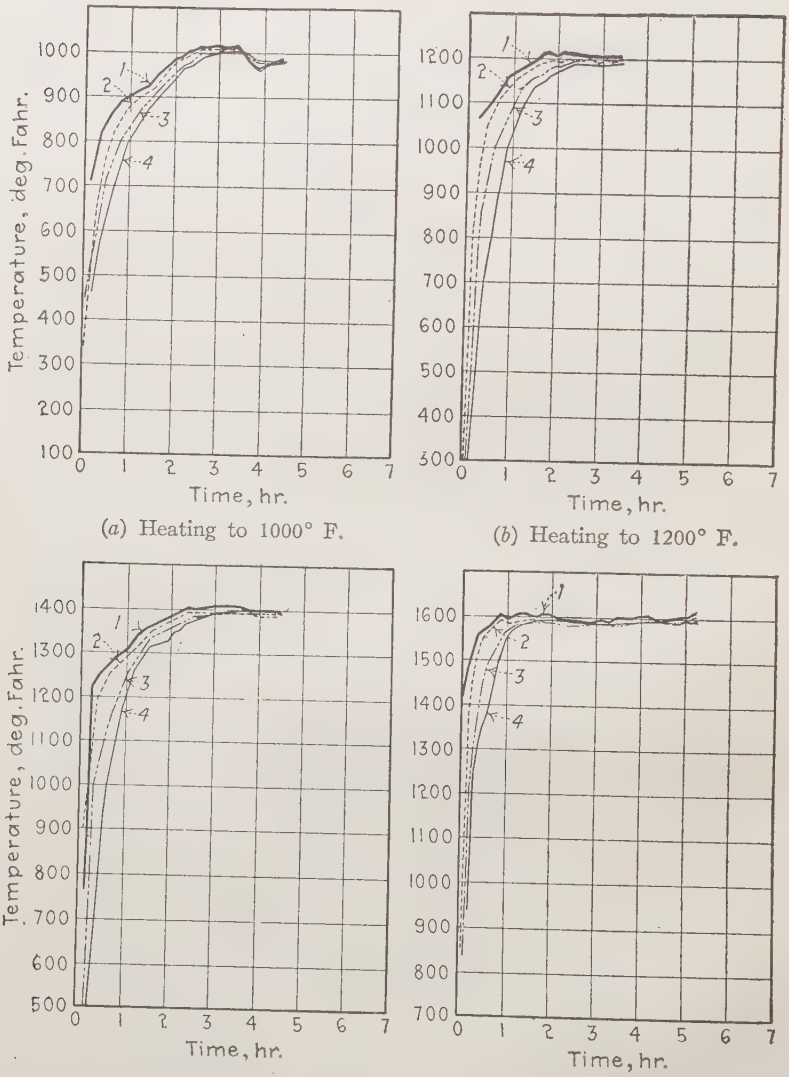
(*c*) Heating to 1400° F.

(*d*) Heating to 1600° F.

LEGEND.

1....Upper furnace couple. 3....Contact couple.
2....Lower furnace couple. 4....Couple at center.
6....Couple 2 in. from surface.

FIG. 6.—Rates of Heating 8-in. Specimen.



(*a*) Heating to 1000° F.

(*b*) Heating to 1200° F.

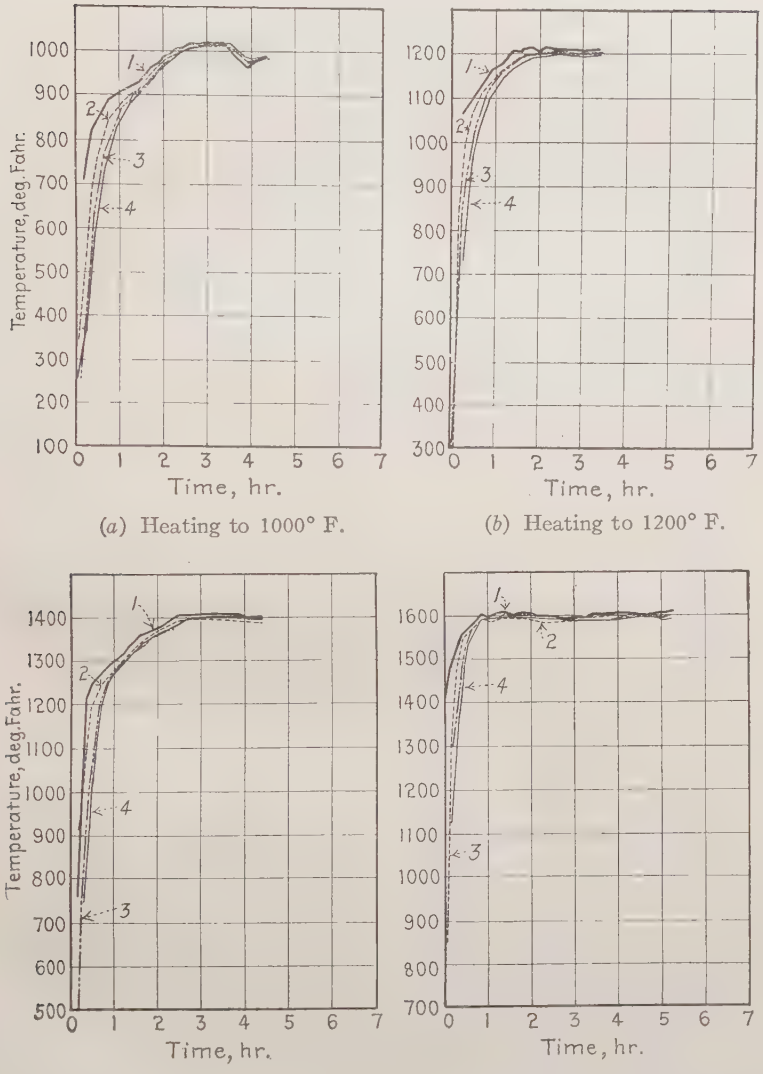
(*c*) Heating to 1400° F.

(*d*) Heating to 1600° F.

LEGEND.

1....Upper furnace couple.
2....Lower furnace couple.
3....Contact couple.
4....Couple at center.

FIG. 7.—Rates of Heating 4-in. Specimen.



(*a*) Heating to 1000° F.

(*b*) Heating to 1200° F.

(*c*) Heating to 1400° F.

(*d*) Heating to 1600° F.

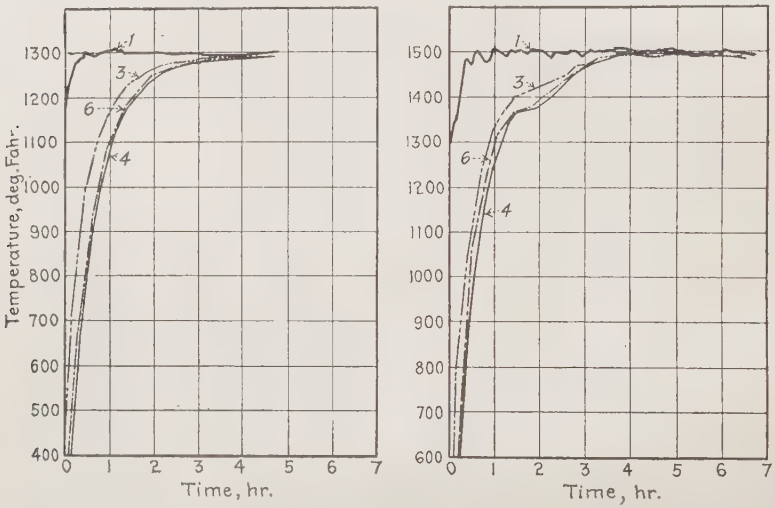
LEGEND.

1....Upper furnace couple. 3....Contact couple.
2....Lower furnace couple. 4....Couple at center.

FIG. 8.—Rates of Heating 2-in. Specimen.

the constantan wires. For convenience in making the connections, as well as for the purpose of minimizing conduction errors, the constantan was not welded directly to the full-size piece of iron wire, but to a small piece split off from it, as shown in Fig. 10.

The contact couple had its welded junction exposed and was held in contact with the surface of the specimen. The



(*a*) Heating to 1300° F. (*b*) Heating to 1500° F.

LEGEND.

1....Furnace couple. 4....Couple at center.
3....Contact couple. 6....Couple 2 in. from surface.

FIG. 9.—Rates of Heating 8-in. Specimen.

contact was quite well shielded, however, from the direct action of furnace gases by ceramic insulators.

CONCLUSIONS.

Four interesting conclusions may be drawn from these experiments.

1. *Variation in Time of Heating with Size.*—As would be expected, the smaller specimens heat more rapidly than the larger. In the curves shown in Fig. 11, the relations between

the size of specimen and time of heating to various temperatures are brought out, the data being taken from Figs. 5 to 9, inclusive. Except in a very general way, this information could not be used as a guide to heating practice, as the rates would vary with the size of furnace and probably with other conditions.

2. *Relation Between Time of Heating and Furnace Temperature.*—It will be noted from Fig. 11 that the time of heating for a specimen of any size is less when it is brought up to 1600° F. than when brought up to 1200° F. and less for 1200° F. than for 1000° F., although it is greater for 1400° F. than for any other temperature.

The fact that it takes longer to heat to 1400° F. than to any other temperature is readily explained. The material experimented on was carbon steel, having its transformation point at about 1350° F. As will be seen from the curves of

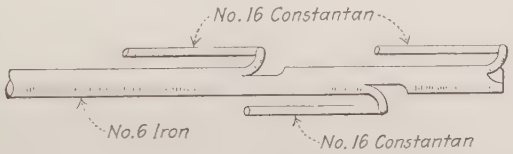


FIG. 10.—Construction of Multiple Thermocouple.

center temperatures of the 12-in. and 8-in. specimens, these specimens hung at the critical temperature for quite a long time—over an hour in the case of the 12-in. specimen. Evidently the large amount of heat absorbed by the steel in carrying it through its transformation point, makes it necessary to allow a long time to raise it above this point when the furnace temperature is only slightly above it.

The longer time required to heat each specimen up to 1400° F. is clearly brought out in Fig. 12, in which time is plotted against temperature, with a curve for each size. The additional runs on the 8-in. specimen at 1300 and 1500° F. were made to see how the time of reaching these temperatures compared with the others, and the results are shown on the curve for the 8-in. specimen in Fig. 9.

It is more difficult to account for the fact that the higher temperatures are attained more rapidly than the lower ones.

This fact, however, appears to be clearly demonstrated. It may be that the specimens received a large amount of their heat by radiation from the furnace walls. The heat transfer by radiation between two bodies at different temperatures is proportional to the difference between the fourth powers of their absolute temperatures, and so for a 100° difference in temperature between furnace wall and test specimens, at 1600° F., the heat transfer

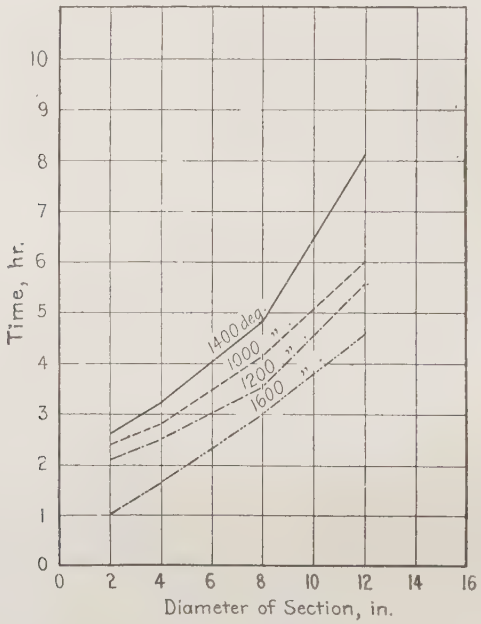


FIG. 11.—Variation of Time of Heating with Size of Specimen.

would be at a higher rate than for the same temperature difference at lower temperatures.

In Fig. 13, the curves for the heating of the centers of the 12-in. and 8-in. specimens to the furnace temperatures, namely, 1000, 1200, 1400 and 1600° F., are superimposed, and bring out the relations discussed above more clearly than do the separate curves. These curves have been idealized to the extent of smoothing out the irregularities of the furnace temperatures and those of the specimen supposed to be due to furnace irregularities.

3. *Relations Between Surface and Interior Temperatures.*—From all of the curves, it will be noted that there is no large difference in temperature of the points inside of the specimen. This was quite surprising, as it was expected that the 12-in. specimen would show considerable differences of temperature between a point 2 in. from the surface and the center.

It might be thought that the type of multiple couple shown in Fig. 10 would be subject to errors due to conduction along its

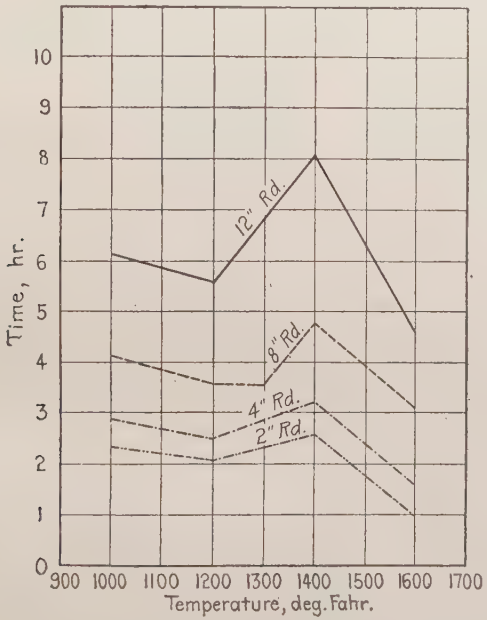


FIG. 12.—Variation in Time of Heating with Temperature of Furnace.

common iron wire, but this point was carefully investigated before it was used, by comparing its readings with those of independent couples at the same depth in an iron pipe, heated to different temperatures along its length, and it was found free from such errors. If there were errors due to conduction from the outside, they would exaggerate rather than minimize the differences. The possibility of all parts of the hole being brought to the same temperature by air circulation was eliminated by filling the space around the couple with asbestos.

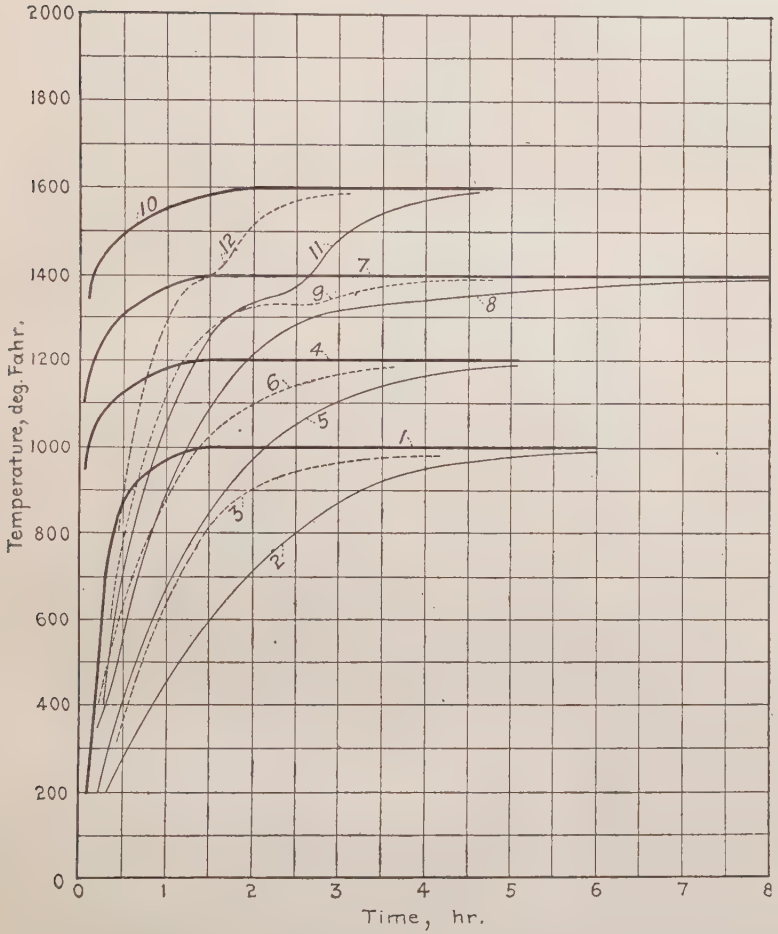
As would be expected, there is a greater difference at rapid rates of heating than at slow rates of heating.

All of the runs show that the contact couple is at a higher temperature than any of the interior couples until the specimen has attained the temperature of the furnace. It cannot properly be assumed that the temperature shown by the contact couple is exactly that of the surface of the specimen. Although it was attempted to guard the junction from the direct influences of the furnace gases, it is altogether probable that it was somewhat influenced by them, and that the temperatures which the contact couple indicates are higher than even the outermost layer of the material.

It will be noted from all of the curves that when the contact couple attains the furnace temperature, all parts of the specimen have also attained that temperature. This suggests a practical method of using contact couples in conjunction with furnace couples, namely, by means of the furnace couple the furnace should be held at the temperature at which it is desired to treat the specimen, and the contact couple should then be used to determine when the specimen has assumed the desired temperature.

4. *Contact Couple Shows Time of Transformation.*—It will be observed from the curves showing the heating of the 12-in. and 8-in. specimens to 1400 and 1600° F. that the transformation point is clearly shown by the couples inside of the specimen, and that it is also shown by the contact couple. The interior couples show, with approximate correctness, the temperature at which the transformation takes place. The contact couple shows a corresponding flexure in its curvature, at the same time as the interior couples, though not at the same temperature. The close correspondence in time between the flexures of the contact couple and the interior couples, points to what the writer believes is an important new method of determining when a piece of steel has been heated through its transformation point.

The contact couples of the 2-in. and 4-in. specimens do not show this phenomenon so clearly. This is due to two conditions of the experiment. The record was made by a 6-point recorder, and as a result an interval of six minutes elapsed between succes-



HEATING TO 1000° F.

1....Furnace temperature.
2....12-in. specimen.
3....8-in. specimen.

HEATING TO 1200° F.

4....Furnace temperature.
5....12-in. specimen.
6....8-in. specimen.

HEATING TO 1400° F.

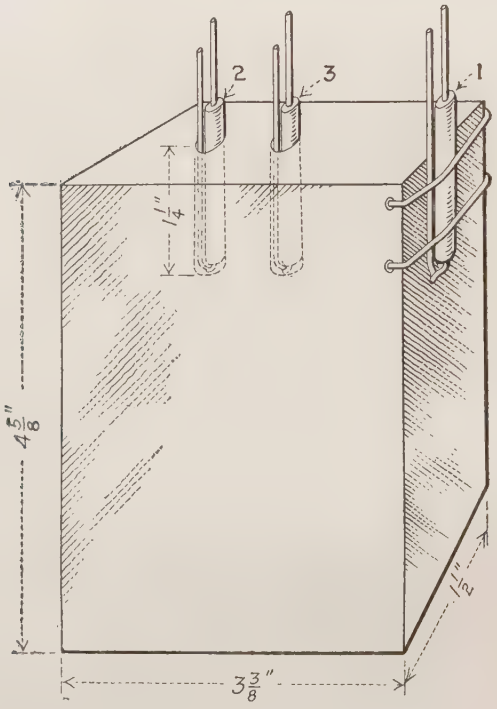
7....Furnace temperature.
8....12-in. specimen.
9....8-in. specimen.

HEATING TO 1600° F.

10....Furnace temperature.
11....12-in. specimen.
12....8-in. specimen.

FIG. 13.—Rates of Heating Centers of 12 and 8-in. Specimens to Furnace Temperatures.

sive points on each record. With small pieces which go through the transformation point rapidly, the curve must be nearly a continuous one to show the phenomenon. In order to bring it out clearly, it is also important that the rate of heating be a uniform one. If the rate is not uniform, there will be irregulari-



LEGEND.

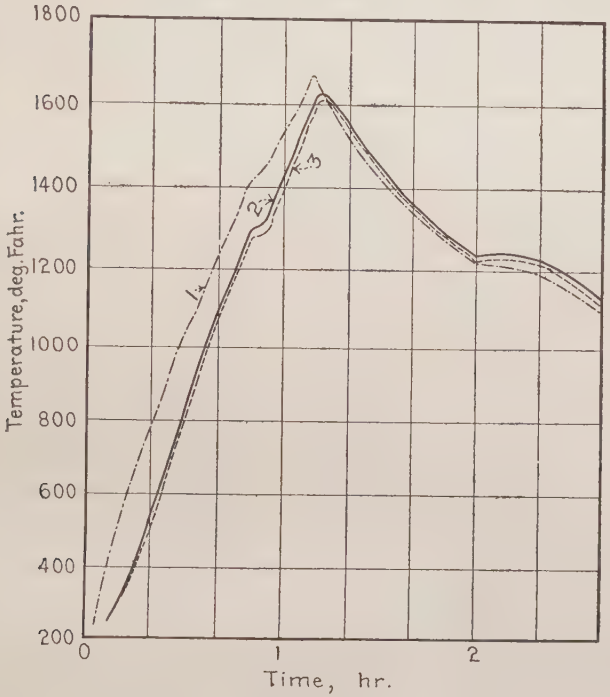
1....Contact couple. 2....Couple 2 in. from surface.
3....Couple at center.

FIG. 14.—Arrangement of Thermocouples on Small Specimen.

ties in the contact-couple curve which may mask or resemble the flexure due to the critical point, and lead to much uncertainty in interpreting it. In Fig. 6, showing the heating of the 8-in. specimen to 1000° F., such a flexure is clearly shown, and it is obviously due to a drop in the furnace temperature. Many runs have been made on small specimens, with furnaces that

permitted uniform rates of heating, and have regularly shown the flexure of the contact-couple curve at the time of transformation.

In Fig. 14 is shown the arrangement of three couples, one at the surface and two in the interior of a small block of steel.



LEGEND.

1....Contact couple. 2....Couple 2 in. from surface.
3....Couple at center.

FIG. 15.—Heating and Cooling Curves of a Small Specimen.

This specimen was heated uniformly in an electrical furnace, and the temperature of each couple recorded on a curve-drawing recorder, the charts of the three recorders having been carefully synchronized. The temperature curves of the three couples, both for heating and cooling, are shown in Fig. 15. From this it will be seen that even with the small block of steel there is a

distinct flexure in the contact couple at exactly the time that the interior couples show the specimen to be passing through its transformation point.

This method of determining the time of transformation of a piece of steel has been used under the writer's observation for hardening a large number of punches and dies, as well as other small pieces, and has resulted in exceptional uniformity and high quality of product. The pieces have not only come out properly hard, but have also been to a very unusual degree free from deformation and cracks. It may be pointed out in passing that this method of determining when the piece has been properly heated does away with the necessity of a prior determination of the critical point on a small sample, and also entirely does away with the necessity of a painstaking calibration of the apparatus. The contact couple is not used to determine the temperature of transformation, but the time of transformation, and it is therefore of relatively little importance whether it is accurately calibrated or not, so long as it is properly sensitive.

Experiments have shown that this method may be used with quite small pieces, even as small as the end of the thermocouple.

The customary note of thanks to those who have assisted in the work would in this case be quite inadequate. The work was actually done by Mr. Radclyffe Furness and Mr. A. H. Miller, of the Midvale Steel Co., and Mr. William J. Wrighton of the Leeds and Northrup Co. The writer can only claim whatever credit may attach to having encouraged the work and presented the results.

DISCUSSION.

Mr. G. K. Burgess (*presented in written form*).—The results here presented by Mr. Leeds are of the greatest interest and importance and constitute a very positive advance in the available knowledge of the inter-relations of the four principal factors met with in heating steel, namely, the temperature of furnace, and the size, temperature distribution and time to attain uniformity of temperature for the specimen. Mr. Burgess

The experiments are also particularly of value in that they have been carried out with specimens of wide variation in size, or from 2 to 12-in. rounds, so that the results obtained are readily and immediately applicable in practice.

The writer should also like to emphasize the evident advantages of the kind of cooperation of which this paper is such an excellent example, namely, between the technical personnel of a manufacturing plant or laboratory and the instrumental expert. There are undoubtedly many other similar instances where this sort of cooperation would lead to more valuable results than can normally be expected to be obtained by either alone.

Mr. H. V. Wille.—The experiments of Mr. Leeds are very instructive. As I interpret them, they show that under ordinary commercial methods of heating, there is a difference of temperature of about 10° between the inside and outside of a 12-in. forging. About 1908, I made a similar experiment with a Bristol pyrometer, and these experiments also showed that there was a very small variation between the inside and outside of a forging when it was being heated. The experiments also show the inconsistency of some of our specifications for forgings. The specifications, especially those for heat-treated forgings, provide that the forging shall be slowly and uniformly heated. The men in charge of the heat treatment and of the annealing of forgings, are very careful to take every possible precaution to prevent any great difference of temperature between the interior and exterior of the forging. A simple calculation based Mr. Wille.

Mr. Wille. upon the heat conductivity of steel, will show that under usual manufacturing conditions there is little danger to be apprehended from this cause, since the commercial rate of heating is usually much slower than the rate of transmission of heat through steel. It usually takes about an hour and a half to three hours to heat a forging, and we carefully specify that it shall be slowly and uniformly done. But then we specify a tensile strength, elastic limit, elongation and reduction of area which require the manufacturer to cool the forging very rapidly, and instead of taking from one and a half hours to three hours to cool it, the specified requirements may necessitate cooling through the critical range in two to three minutes and sometimes even more quickly, thus producing a lag, or difference in temperature, of 500 or even 1000°, with correspondingly high internal stresses. All the good work, having in view the prevention of internal stresses and cracks, which we expect to accomplish by slowly heating the forging, is set at naught by this rapid cooling.

At the same time that I made experiments to determine the difference between the temperature of the inside and the outside of the heated forging, I also made experiments to determine the difference of temperature between the inside and outside of a cooled forging of 12 in. diameter, and secured differences between 500 and 1000°. It was on account of obtaining such high differences of temperature, that I cut deformation rings from the forgings and determined the stresses.[1] At the present time, I have under way a series of experiments to determine the difference of temperature between the inside and the outside of a cooled forging under different rates of cooling, this being just about the reverse of the experiments conducted by Mr. Leeds. I expect to have this work completed in a month or two, and I hope to be able to present the results to the Society at the next annual meeting.

Mr. Abbott. MR. ROBERT R. ABBOTT.—I wish to express my appreciation of this paper. It is extremely interesting. I should like to discuss briefly one of the points considered by Mr. Wille in his remarks. It is very true that apparently we are inconsistent

[1] H. V. Wille, "Internal Stresses Developed by Different Quenching Mediums and Their Effects," p. 27.—ED.

in demanding, or rather desiring, that we raise the temperature very slowly in large forgings, and that we should then turn around and apparently work in the opposite way by requiring a rapid cooling. However, upon second thought, there is no real inconsistency, because in heating a forging the outside will be hotter than the interior and the tendency is a tearing apart, because this hot outside is attempting to pull itself away from the cooler inside. Thus there is a tendency to develop cracks at any place where there is a change in section, or a sharp corner. Upon quenching, however, we have exactly the opposite effect, the outside is being cooled rapidly and is colder than the inside, and the tendency is towards compression. I have had a great deal of experience in heating and heat-treating large forgings, and I know that the secret of success, particularly with intricate dies, is in slow heating. Mr. Abbott.

MR. RADCLYFFE FURNESS.—There is one point that I think has been overlooked in Mr. Wille's remarks as to our reversal in heat treatment. When a forging is heated, the aim is not only to get the carbon in the forging in solution, but also to diffuse the dissolved carbon throughout the mass so that there may be a homogeneous solution. In quenching, however, the contrary aim is to stop all carbon from coming out of solution, and therefore the cooling should be done as quickly as possible. That is, the practice is reversed in order to bring about reverse action. Mr. Furness.

MR. WILLE.—I agree with Mr. Abbott that rapid heating will induce tensile stresses in forgings, which, of course, renders them more prone to fracture than if the stresses were compressive. On the other hand, rapid cooling induces compressive stresses in forgings, provided the rate of propagation of heat from the interior to the exterior of the forging is equal to the rate of conduction of heat from the surface. If there is a lag in the temperature, both tensile and compressive stresses may be induced by cooling. In fact, the forging after rapid cooling is in a state of stress similar to that existing in a Prince Rupert drop, so that even a slight shock may result in fracture. Mr. Wille.

MR. K. W. ZIMMERSCHIED.—Mr. Leeds points out that an uncalibrated couple on the surface of the specimen will indicate the time at which the steel goes through its critical range, and, Mr. Zimmerschied.

Mr. Zimmerschied.

by inference, I take it that he intends this to be an indication of the time at which the quenching should be done. That is true if the piece has previously had a diffusion anneal, that is, an anneal in which the carbides have had a chance to diffuse throughout the whole specimen uniformly, but unless such previous heat treatment has been given, as in a case where we endeavor to get a sample of uniform structure throughout on one quench, this procedure will not be feasible. It is necessary to heat-treat steel of moderate carbon content very considerably above this halting point before a uniform solution results. Therefore, this halt would have no significance for a "single quench," which is, I venture to say, by far the most widely used procedure in commercial work.

Mr. Leeds.

MR. M. E. LEEDS.—In closing, I have very little to say except to call attention to the limitations of the paper. Referring to Mr. Wille's remarks, for instance, that it showed that under commercial conditions the temperature variation inside the piece is very small: the paper shows that under certain rates of heating, and with certain specimens, the temperature variation is small. That is probably generally true for a considerable range of rates of heating, and sizes of specimens, but nothing should be inferred about what the temperature gradient may be for rapid rates of heating.

Regarding Mr. Zimmerschied's statement that it might be inferred that the method was intended to show at what temperature specimens should be quenched, I am very innocent of expert knowledge in regard to the heat treatment of steel, and I certainly did not intend that any use of the contact couple for determining the temperature at which to quench the specimens should be made except; that where it is the aim of the treatment to quench the specimen just above the critical temperature, this method may be used for finding when the specimen has reached that temperature.

INTERNAL STRESSES DEVELOPED BY DIFFERENT QUENCHING MEDIUMS AND THEIR EFFECTS.

By H. V. Wille.

SUMMARY.

There are no data available on the internal stresses resulting from quenching in different mediums and under different conditions, and these stresses are determined in different portions of a cylinder about 9½ in. in diameter, quenched in water, light tempering oil, heavy tempering oil, fish oil and cottonseed oil. A slow and mild water quench results in a fiber stress of 45,000 lb. per sq. in.; light tempering oil about 35,000, heavy tempering oil about 24,000, fish oil about 30,000 lb. per sq. in.

The physical properties of steel quenched under these conditions are also given, and show that the tensile properties of rapidly cooled steel are superior to those cooled at a slower rate. The effect of an extremely high rate of cooling is illustrated in a photograph (Fig. 5) showing internal cracks in a carbon-steel forging cooled at an extremely high rate, but a very high elastic limit, elongation and reduction of area resulting therefrom.

The results of the experiments show that metallurgists are confronted with the alternative of being satisfied with conservative physical properties in quenched-and-tempered material, or of accepting the danger of an occasional failure from the development of internal fracture.

The effect of the initial temperature of the quenching medium on tensile properties is shown to be of no serious moment, but the internal stresses are affected thereby.

INTERNAL STRESSES DEVELOPED BY DIFFERENT QUENCHING MEDIUMS AND THEIR EFFECTS.

By H. V. Wille.

The exact nature of the changes which take place within the critical range, or at the three transformation points of steel, is still a subject of discussion, but it is a matter of common knowledge that profound changes do take place, changes so profound that all of the physical properties are affected. Within this range steel undergoes the following:

1. Loss of the property of magnetism;
2. A pronounced evolution of heat;
3. A distinct change in the rate of increase in electric conductivity;
4. A curious and abrupt change in the linear dimensions;
5. The tensile strength no longer falls with an increase in temperature, but suddenly increases.

These changes all take place at approximately the same temperature, in grades of steel with sufficient carbon to warrant quenching them for the purpose of improving their tensile strength and elongation, and the coincidence of these changes in properties is almost perfect at the critical temperature of an eutectic steel.

These remarkable changes have been the subject of investigation and study by the most eminent physicists. The microstructure of steel of different compositions, quenched at the critical temperature, has also been the subject of much research, and the art has now reached a stage that the trained metallographist can predict in general terms and with reasonable accuracy, the microstructure of steel of different carbon contents, provided the specimens are small and the quenching is sufficiently rapid to retain the molecular structure induced by heating to the critical temperature.

The effect of quenching and annealing on the tensile strength, elongation and reduction of area of different grades of steel, is also well understood, and there is a large mass of data available giving the tensile properties for various quenching conditions.

Both the microstructure and the tensile strength, however, are affected by the rate of cooling, and the rate of cooling is in turn governed by the cooling power of the quenching medium and the mass of the quenched objects. The surface of the quenched objects may also influence these properties; Benedicts' experiments, however, indicate that the surface has little influence.

The classical work of Benedicts[1] demonstrates that the quenching power of a liquid depends largely on the latent heat of vaporization, and refutes the views commonly held that it depends upon the initial temperature or the heat conductivity of the medium. The addition of salt to increase the quenching power of water is also proved fallacious, and the experiments of

TABLE I.—FIBER STRESS DUE TO RAPID COOLING.

Difference of Temperature, deg. Fahr.	Fiber Stress, lb. per sq. in.
1500	270 000
1000	180 000
500	90 000
100	18 000

Le Chatelier that the conductivity for heat is no measure of the cooling power of a liquid are confirmed.

Mercury has a heat conductivity of over ten times that of water, but both of these able physicists demonstrate that it is decidedly weaker as a cooling medium. Le Chatelier considers that the specific heat of the cooling medium governs the cooling speed,—and this is confirmed by Benedicts,—providing the temperature of the body is low, but the latent heat of vaporization is the controlling factor when the temperature of the body is high. This theory is tenable considering the large amount of heat which can be carried off by a relatively small weight of vapor, and accounts for the efficiency of water as a quenching medium.

Water is the oldest known cooling medium for quenching steel. Subsequently, various animal and vegetable oils were

[1] "Experimental Research on the Cooling Power of Liquids and on Quenching Velocities."

employed, and many of the artisans using them are still under the belief that carbon is added to the steel by these liquids and the properties thereby improved. In this country mineral oil, because of its low cost, has almost entirely displaced the animal and vegetable oils, and for many years was used almost exclusively for quenching spring plates and large objects. Water, however, possesses manifest advantages of cheapness, cleanliness, freedom from odor, freedom from fire hazard, and above all, efficiency. It is true that a higher elastic limit, tensile strength and elastic ratio for a corresponding elongation and reduction

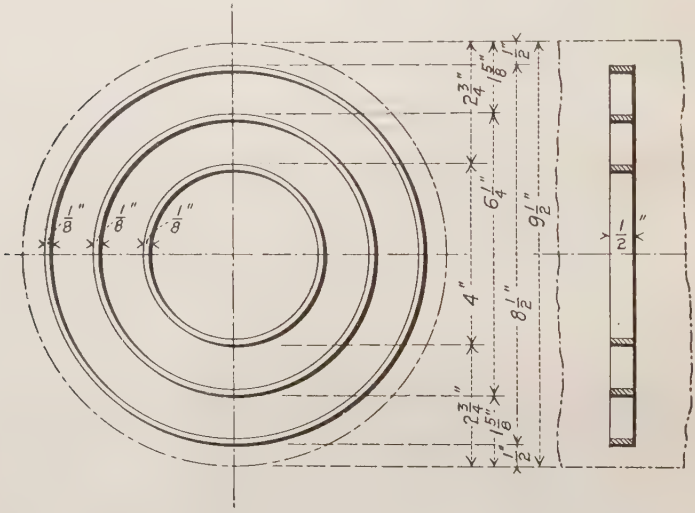


FIG. 1.—Location of Stress-Deformation Rings Cut from Forging.

of area, or a higher reduction of area and elongation for a corresponding elastic limit, can be obtained on a water-quenched steel than can be secured by the methods of oil quenching generally practiced. Opposing these manifest advantages is the danger from fractures as the result of the lag in the temperature of the steel.

Table I shows the fiber stress induced by the difference in temperatures between the inside and outside of a heated piece of steel, subjected to rapid cooling.

These figures were so startling that several years ago the

author made a number of experiments to determine the internal stresses in oil and water-quenched forgings of diameters from 6 to 12 in. In recent years many different grades of oil have been proposed as being more effective than the high flash-test mineral oil generally employed, and which could be used without danger of producing a fracture in the steel, and the experiments were extended to include such oils. Full-size specimens, $9\frac{1}{2}$ in. in diameter, cut from a driving axle, were employed, and a conservative method of cooling was followed such as is used in practice, since it was not the intention to determine the maximum stress obtainable, but simply those which would result under usual commercial conditions of quenching. The following quenching mediums were employed:

Water		
Light tempering oil	Specific Gravity	30.9 B.
	Flash Point	400° F.
	Fire Test	445° F.
	Viscosity	112 seconds at 100° F.
Heavy tempering oil	Specific Gravity	25.6 B.
	Flash Point	550° F.
	Fire Test	625° F.
	Viscosity	178 seconds at 212° F.
Fish oil	Specific Gravity	23.3 B.
	Flash Point	405° F.
	Fire Test	440° F.
	Viscosity	165 seconds at 100° F.
Cottonseed oil	Specific Gravity	22.6 B.
	Flash Point	580° F.
	Fire Test	630° F.
	Viscosity	160 seconds at 100° F.

The forgings were heated to 1550° F. and quenched in the foregoing mediums at initial temperatures of 70, 150, 212, 300 and 400° F. Observations were made of the rise of the temperature of the quenching medium and of the time of immersion required to cool the forgings to 600° F.

A series of three rings was then cut from each section as shown in Fig. 1. These rings were accurately micrometered before they were detached and were again accurately measured after detachment from the forging and the fiber stress calculated

from the deformation of the rings. From Fig. 1 it will be noted that $\frac{1}{2}$ in. of metal was first removed so that the first ring represents the fiber stress $\frac{1}{2}$ in. from the surface of the forging, the second ring gives the result $1\frac{5}{8}$ in. from the surface and the third ring $2\frac{3}{4}$ in. therefrom. The various detailed measurements are of no particular value and will not be given. The detached ring in all cases increased in diameter, showing that $\frac{1}{2}$ in. under the surface the fibers were stressed in compression.

The explanation is simple. If a cylindrical object is heated to 1500° F. and suddenly quenched in some cooling medium, the surface contracts so that the outer fibers are in tension and the inner fibers in compression. This is shown in Fig. 2, the outside or cross-hatched portion shrinking on the interior portion, so that the outside is in tension and the inside in compression. It is

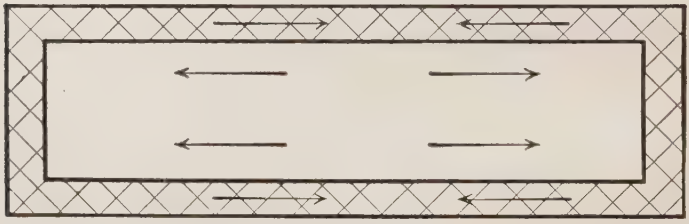


FIG. 2.—Stresses in a Quenched Cylinder.

interesting to note that the volume of all the cylinders was decreased by quenching. The fiber stresses induced by the different quenching mediums are given in Table II.

Table II shows that the lowest fiber stresses are induced by the heavy tempering oil very generally used, and these stresses are never above the elastic limit of the material. The greatest fiber stresses are induced by water, and the immersion temperature appears to have but little influence in ameliorating the internal stresses. It must also be remembered that the pieces were not rapidly cooled in the water, and that they were withdrawn when they attained a temperature of about 600° F. so that the stresses were reduced by slow cooling from this temperature. Stresses can be produced by water quenching equal to the calculated stresses given in Table I, simply by increasing the

rate of cooling. It would be unsafe to increase these stresses beyond those produced in the water-quenched specimens, notwithstanding the fact that the tensile properties would be increased thereby.

TABLE II.—FIBER STRESSES INDUCED BY DIFFERENT QUENCHING MEDIUMS.

Temperature of Medium, deg. Fahr.		Time of Immersion, minutes.	Fiber Stress, lb. per sq. in., at distance from surface of		
Before Quenching.	After Quenching.		$\frac{1}{2}$ in.	$1\frac{1}{8}$ in.	$2\frac{3}{4}$ in.
Quenched in Water.					
70	210	13	42 600	29 500	16 500
150	boiling	22	46 800	15 300	8 100
212	boiling	22	40 200	51 000	15 700
Quenched in Light Tempering Oil.					
70	330	12	42 300	16 800	4 500
150	374	21	40 500	10 800	2 800
212	435	22	17 650	15 300	4 600
300	410	28	12 890	12 990	6 000
400	fire	23	18 330	16 200	.....
Quenched in Heavy Tempering Oil.					
70	330	12	24 000	5 100	6 250
150	405	22	23 500	16 200	10 500
212	400	22	17 400	600	750
300	445	30	24 000	2 500	11 600
400	460	30	2 460	.....	9 750
Quenched in Fish Oil.					
70	334	12	39 200	26 100	15 000
150	389	20	23 500	16 200	10 500
212	440	21	27 900	9 090	11 250
300	430	25	30 600	13 980	15 100
400	428	30	29 100	10 560	21 750
Quenched in Cottonseed Oil.					
70	338	12	30 300	17 700	12 000
150	375	18	25 410	4 200	15 600
212	440	22	26 800	11 100	15 000
300	440	25	19 840	18 240	18 600
400	448	25	16 230	12 480	.....

In order to determine the advantages resulting from rapid cooling, test bars were cut from each one of the specimens, and tested (1) without any annealing and (2) after drawing to 1150° F. The results of these tests are shown in Table III.

TABLE III.—PROPERTIES OF TEST BARS CUT FROM SPECIMENS, QUENCHED IN DIFFERENT MEDIUMS.

QUENCHED IN WATER.

Temperature of Medium, deg. Fahr.		Time of Immersion, minutes.	Elastic Limit, lb. per sq. in.		Tensile Strength, lb. per sq. in.		Elongation in 2 in., per cent.		Reduction of Area, per cent.	
Before Quenching.	After Quenching.		Annealed.	Unannealed.	Annealed.	Unannealed.	Annealed.	Unannealed.	Annealed.	Unannealed.
70	210	13	55 560	70 440	95 960	105 400	23.0	16.0	41.9	30.0
150	boiling	22	55 940	68 160	90 600	100 500	25.0	20.0	46.5	35.8
212	boiling	20	56 160	69 730	102 650	107 850	23.0	19.0	47.3	25.5
		Aver....	55 900	69 443	96 400	104 583	24.0	18.3	45.2	30.4

QUENCHED IN LIGHT TEMPERING OIL.

70	330	12	52 550	58 130	87 750	89 160	29.0	25.0	50.5	45.8
150	374	21	53 760	64 000	92 460	97 500	23.0	19.0	43.7	38.0
212	435	22	59 710	57 000	98 060	103 000	21.0	16.0	36.4	26.0
300	410	28	54 540	64 680	98 480	101 500	24.0	20.0	38.4	33.3
400	on fire	23	55 280	71 000	89 460	100 000	26.0	20.6	48.2	34.0
		Aver....	55 200	62 962	95 200	98 232	24.6	20.1	43.6	35.4

QUENCHED IN HEAVY TEMPERING OIL.

70	330	12	53 330	55 340	82 240	91 380	31.0	25.0	53.8	44.7
150	405	22	55 820	63 080	91 050	99 480	21.0	18.0	42.0	28.2
212	400	22	54 820	63 160	90 360	94 260	26.0	21.0	44.7	36.8
300	445	30	55 380	69 560	99 480	99 040	22.0	20.0	37.9	34.8
400	460	30	52 000	59 610	89 000	91 640	25.0	23.0	42.0	39.9
		Aver....	53 300	62 150	90 400	95 160	25.0	21.4	44.3	36.8

QUENCHED IN FISH OIL.

70	334	12	57 490	64 500	93 240	100 000	25.0	21.0	46.9	38.0
150	389	20	53 300	67 180	91 380	103 600	25.0	17.0	44.7	29.2
212	440	21	53 840	67 840	90 790	103 000	24 0	19.0	44.1	31.7
300	430	25	57 480	63 060	91 790	95 080	24 0	23.0	43.0	40.4
400	428	30	63 960	67 500	103 500	107 500	17.0	15.0	27.9	22.7
		Aver....	57 200	66 016	92 100	101 856	23.0	17.0	41.3	32.4

QUENCHED IN COTTONSEED OIL.

70	338	12	54 550	62 500	92 930	97 500	23.0	21.0	44.4	37.0
150	375	18	49 490	54 460	86 890	91 090	26.0	22.0	48.0	35.2
212	440	22	49 040	56 220	89 420	94 520	24.0	22.0	43.7	38.8
300	440	25	57 000	67 000	99 500	102 950	22.0	19.0	39 1	31.5
400	448	25	52 600	63 370	95 740	101 500	22.0	19.0	38 9	29.2
		Aver....	52 100	60 710	92 800	97 512	23.6	20.6	42.8	34.3

The analysis of the steel is as follows:

Carbon	0.41	per cent
Manganese	0.55	"
Phosphorus	0.036	"
Sulfur	0.024	"
Silicon	0.023	"

It would have been possible to secure tensile properties superior to those obtained by merely increasing the quenching speed, and this is particularly true of the water-quenched specimen, but a more rapid rate of quenching was not followed in

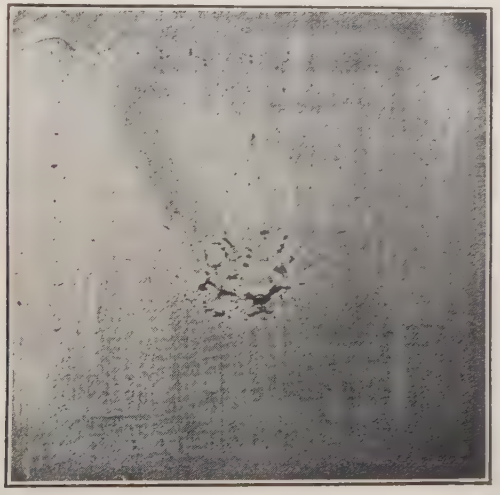


FIG. 3.—Section of Segregated Bloom.

order to insure against the development of a crack in any of the specimens.

These internal strains are the primary cause of internal fractures and their magnitude is clearly dependent on the rate of cooling, but they are aggravated by segregation of impurities or any inequalities in the structure of the steel. Fig. 3 shows a section of such a segregated bloom, and Fig. 4 a section of a forging made from this bloom and then quenched in water. A forging made from a perfect bloom can be fractured, if the rate of cooling is sufficiently rapid. An instance of this kind is

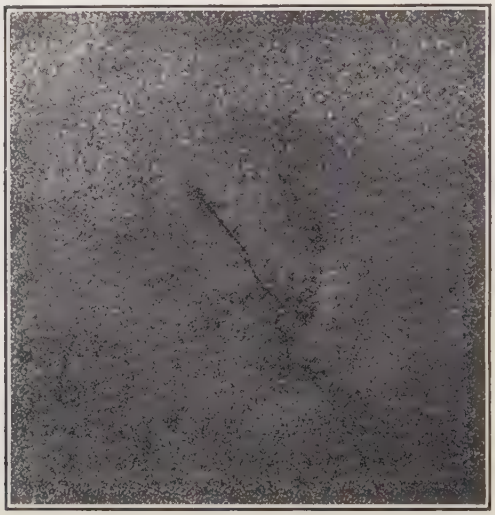


FIG. 4.—Section of Forging Made from Bloom shown in Fig. 3 and Quenched in Water.

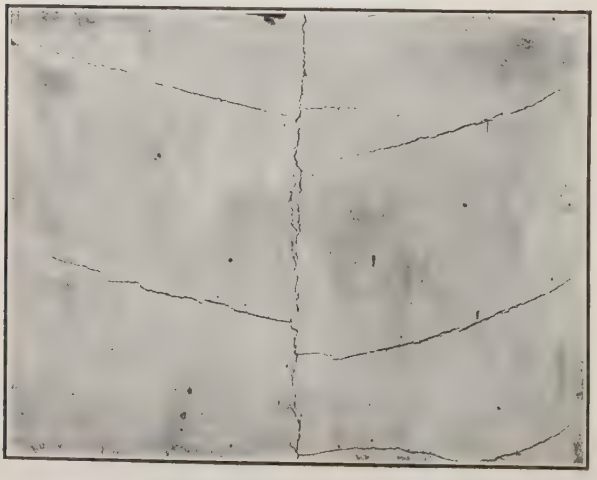


FIG. 5.—Section of Fractured Forging Made from Perfect Bloom.

shown in Fig. 5. A test specimen cut from the forging showed the following remarkable tensile properties:

Elastic limit, lb. per sq. in	57 440
Tensile strength, lb. per sq. in	91 660
Elongation in 2 in., per cent	28.0
Reduction of area, per cent	60.7

The chemical composition of this forging was as follows:

Carbon	0.53	per cent
Manganese	0.43	"
Phosphorus	0.034	"
Sulfur	0.018	"
Silicon	0.01	"
Chromium	none	
Vanadium	none	
Nickel	none	

The rate of cooling also depends upon the chemical composition of the steel, and Benedicts' experiments show that a high-silicon steel has a very much lower rate of cooling than the usual grade of low-silicon steels. It has been observed that the high-silicon steel is more amenable to water quenching than low-silicon steels, but this has been attributed to the fact that such steels are generally denser, more uniform and more free from segregation than the low-silicon steels. The properties no doubt influence the superior results obtained by water-quenching this grade of steel, but the primary cause must be sought in the fact that such steels have a lower rate of cooling, and the internal stresses are therefore less than in a similarly quenched low-silicon steel.

The results of these experiments are as follows:

1. The tensile properties of water-quenched steel are superior to those obtained by quenching in any other of the usual quenching mediums.

2. The internal stresses induced in a water-quenched object are of much greater magnitude than those developed by quenching in any other of the usual quenching mediums.

3. They confirm the laboratory experiments of Benedicts, that the efficiency of the quenching mediums is not dependent to a marked extent on the initial temperature of the cooling mediums.

4. With but few exceptions, which can no doubt be explained by some inequality in the steel, the induced internal stresses are affected by the initial temperature, except in case of water.

5. Internal stresses induced by quenching in water are independent of the initial temperature.

6. The small difference between the temperature of the cooling mediums, before and after quenching, confirms Benedicts' explanation that large quantities of heat are carried off by the latent heat of vaporization.

7. Light oils have a greater quenching speed than heavy oils but not markedly so. A good tempering oil, however, should be free from tar and should not become thick from the precipitation of the burnt tar.

DISCUSSION.

Mr. K. W. Zimmerschied.—Mr. Wille's observation that the ratio of surface to volume in steel is reduced by quenching can be confirmed by an experience we had in quenching a cylindrical piece a large number of times. The cylinder swelled up in the middle and became shortened until it assumed an egg shape, approximating a sphere. Mr. Zimmerschied.

As to the observation that the efficiency of a quenching medium is not dependent to a marked extent on its initial temperature, I believe that this should be qualified to the extent that such initial temperature must be well below the boiling point. If any considerable mass of steel is quenched in water at 200° F. or above, the excessive volume of steam liberated protects the surface and retards the cooling of the metal so far as to actually leave it soft and tough instead of hard and brittle.

Referring to the statement that light and heavy oils do not induce markedly different quenching speeds, we agree in general; in particular cases however, where it is desired to hold maximum hardness, we have found so slight a difference as 25 points in viscosity to result in success or failure. Quenching results were satisfactory with oil of 100 viscosity or less, whereas they were distinctly unsatisfactory when the viscosity was 125 or above, all other conditions being practically the same.

Mr. G. L. Norris.—I notice that there is considerable irregularity in the results in Table II. In a number of cases the fiber stress in the center ring is higher than for the middle ring and even for the outer ring. I should like to ask if there is any explanation for this. Mr. Norris.

Mr. S. V. Hunnings.—I recently had a very striking experience showing the influence of the quenching medium on contraction in diameter when treating shrapnel. When quenching in oil the decrease in diameter amounted to from one to three thousandths; while in water it was as high as eight to ten thousandths. We had hoped that we could save some low-carbon shells by water quenching, but the shrinkage was too great and we have had to use oil exclusively. Mr. Hunnings.

Mr. Wille. MR. H. V. WILLE.—Replying to Mr. Zimmerschied's statement concerning viscosity: it is true that viscosity has some bearing on the efficiency of the quenching medium, but it has the greatest influence with a slow rate of cooling for temperatures under say 800° F. It must be remembered that a cooling medium can cool first by conduction and second by convection. If the cooling is affected by conduction, it is obvious that the specific heat of the medium has an important bearing on the efficiency of the cooling medium; if cooled by convection, and a cooling medium usually operates in this manner as long as no vapors are generated, then of course, the viscosity has a great influence on the cooling effect. I do not think that the viscosity has a marked effect on the efficiency of the cooling medium within ranges of temperature from 1600 to 1000° F. The experiments of both Benedicts and Le Chatelier confirm this statement.

As to the variation shown in Table II, referred to by Mr. Norris, considering the nature of the experiments the variations are not very wide; in fact the results are concordant. Mr. Howard made some determinations of internal stresses in gun forgings some years ago and his results showed variations as wide, if not wider, than those shown in Table II. It should be remembered that in quenching with water, oil, or any other medium, the rate of cooling is not the same in every instance, and that a small difference in the rate of cooling is apt to make a very big difference in the fiber stress.

It should also be remembered that the rate of cooling of a quenched piece is very rapid. If the heat is conducted away from the specimen with greater rapidity than the heat can be propagated from the center of the specimen to the exterior, it is evident that the fiber stresses due to the lag in temperature will be very high. Now if the piece is moved around, or the rate of cooling is more rapid in one case than another, it will make a big difference in the fiber stress.

It should further be remembered that the fiber stresses shown are all within the elastic limit, and for the section shown, the internal ring corresponds to a test piece about 12 in. long; the central ring to one about 18 in. long, and the outside ring to one about 24 in. long. It is obvious that within the elastic limit,

the elastic deformation is very small, and while the micrometers used were accurate to within one-thousandth of an inch, and were estimated within a ten-thousandth of an inch, a very small variation would make a considerable difference in the fiber stress. Mr. Wille.

(*Addendum by letter.*)—I wish to emphasize the general deductions which can be made from these experiments, namely, that very rapid cooling will undoubtedly result in superior physical properties. These results are obtained at the expense of very severe shrinkage strains. The danger attendant upon the development of these strains is evident. The stress per square inch is so near the point of fracture of steel at a blue heat that actual fracture is prevented only by the most careful manipulation and by the most thorough supervision.

For these reasons I have consistently advocated conservative specifications for heat-treated steel, for in my judgment the gain in tensile properties is too small to incur by drastic heat treatment the danger of an internal crack which will surely result in service failure. I have seen a number of failures of heat-treated parts and they have been without a single exception on parts which were rapidly cooled in order to secure tensile properties to conform to some difficult specification.

Before Le Chatelier's or Benedict's experiments were published, the reason for the efficiency of different cooling mediums was not well known. If the medium cooled by conduction, the specific heat would appear to be the controlling factor; if by convection, the viscosity and specific gravity would be of greatest importance; but the results of these experiments indicate that the most important factor which governs the efficiency of a cooling medium is the amount of heat which can be carried off by the vapor. The reason for this is evident. The tensile properties are only affected by a fall through a range from 1600 to 1000° F., and the fall through this range is so rapid that a relatively small amount of heat can be carried away by conduction or convection.

THE RELATION BETWEEN MAXIMUM STRENGTH, BRINELL HARDNESS AND SCLEROSCOPE HARDNESS IN TREATED AND UNTREATED ALLOY AND PLAIN STEELS.

By Robert R. Abbott.

SUMMARY

The object of the investigation was to determine the relation existing between (1) the hardness numbers of steel as determined by the Brinell and scleroscope methods; (2) the maximum strength of steel and the Brinell hardness number; (3) the maximum strength of steel and the scleroscope hardness number.

The investigation was carried out upon three hundred types of steel, each of which was heat-treated in various ways. For this paper the results obtained on carbon, nickel, chrome-vanadium, high-chrome-nickel, and low-chrome-nickel steels only are considered; they comprise 3932 tests.

Eighteen equations have been determined connecting the three quantities, maximum strength, Brinell and scleroscope hardness for the five different types of steel and also for all of the steels considered together.

Comparisons were made between annealed and heat-treated steels, and also between low and high-carbon steels, heat-treated to show the same maximum strength.

THE RELATION BETWEEN MAXIMUM STRENGTH, BRINELL HARDNESS AND SCLEROSCOPE HARDNESS IN TREATED AND UNTREATED ALLOY AND PLAIN STEELS.

By Robert R. Abbott.

The two methods most widely used for the comparison of hardness of steel are the scleroscope and the Brinell. Both methods are so well known that it is unnecessary to enter into a description of them. In general, the scleroscope method gives a measure of the hardness of the surface only, while the Brinell reading is influenced to considerable extent by the condition of the material below the surface.

Formerly, when the maximum strength of commercial steel rarely exceeded 80,000 or fell below 50,000 lb. per sq. in., the testing of the finished material was not so important. However, with the development of modern alloy steels, and particularly in the development of heat treatment of steel, the range in the maximum strength has increased so that we now have steel whose maximum strength varies from 50,000 to over 300,000 lb. per sq. in., and the importance of making accurate determinations of the physical properties of finished material has correspondingly increased. This is commonly done by cutting a test bar for physical tests out of a certain percentage of the finished material. This is an expensive operation, and it becomes particularly so in the testing of steel which has been heat-treated to a strength of over 200,000 lb. per sq. in. Under these conditions it is practically impossible to machine a test bar, and as a result of this, it is becoming more and more customary to measure the hardness of the finished steel and to rely upon the information thus obtained as giving a fair measure of its physical properties.

It was early seen that the hardness test bore some relation to the tensile strength of the steel, but it was also seen that the relation existing between the hardness and toughness apparently

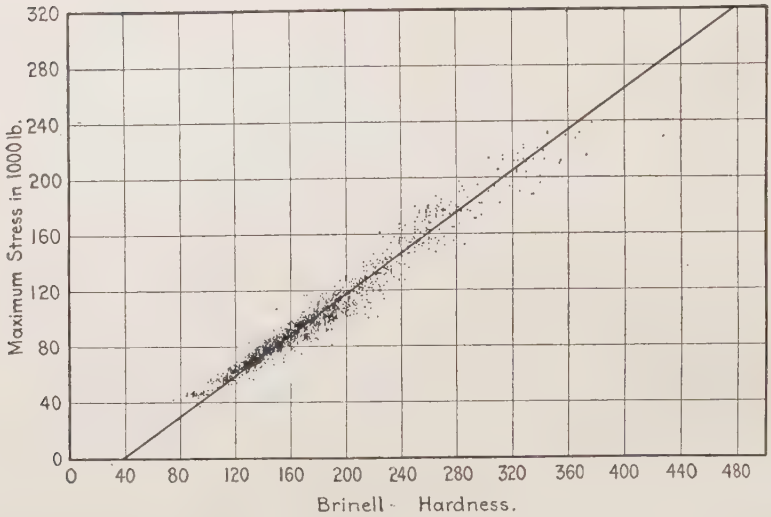


FIG. 1.—Carbon Steels: Relation between Maximum Stress and Brinell Hardness.

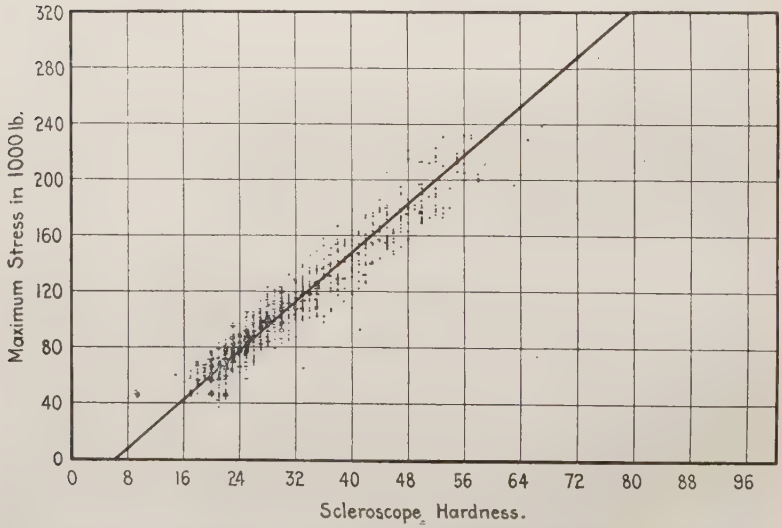


FIG. 2.—Carbon Steels: Relation between Maximum Stress and Scleroscope Hardness.

was not uniform. With a view to obtaining some information regarding the various relations existing among the different physical properties of steel, including the hardness, the following experiments were undertaken about four years ago:

About 300 different types of steel were selected. These included practically all the modern alloy and plain-carbon steels obtainable in this country and abroad. From each steel about 25 test bars were cut. These were machined accurately to $\frac{3}{4}$ in. in diameter and $4\frac{1}{2}$ in. long. The test bars which would be too

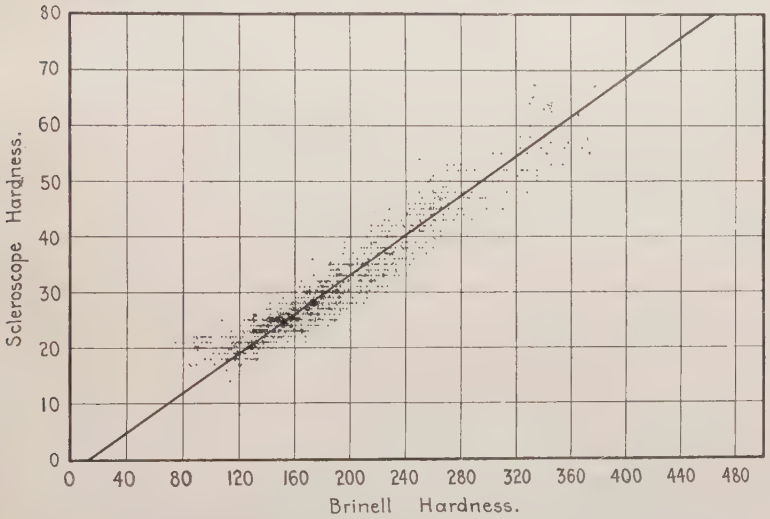


FIG. 3.—Carbon Steels: Relation between Brinell Hardness and Scleroscope Hardness.

hard to machine after treating, were threaded before treatment. All of the critical points of these different steels had previously been accurately determined.

Each test bar was then heat-treated in a different way, to bring out all possible physical properties of that particular steel, and about 5000 were selected for hardness determinations. First, the test specimen was machined according to the standard adopted by the Society,[1] with the exception that one end was left longer than the other by about $\frac{1}{4}$ in., and each test bar was

[1] *Proceedings*, Vol. XII, p. 186 (1912).

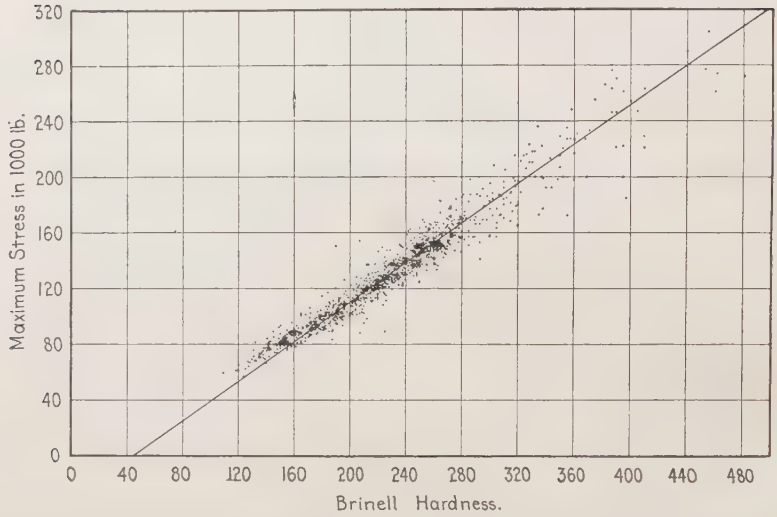


FIG. 4.—Nickel Steels: Relation between Maximum Stress and Brinell Hardness.

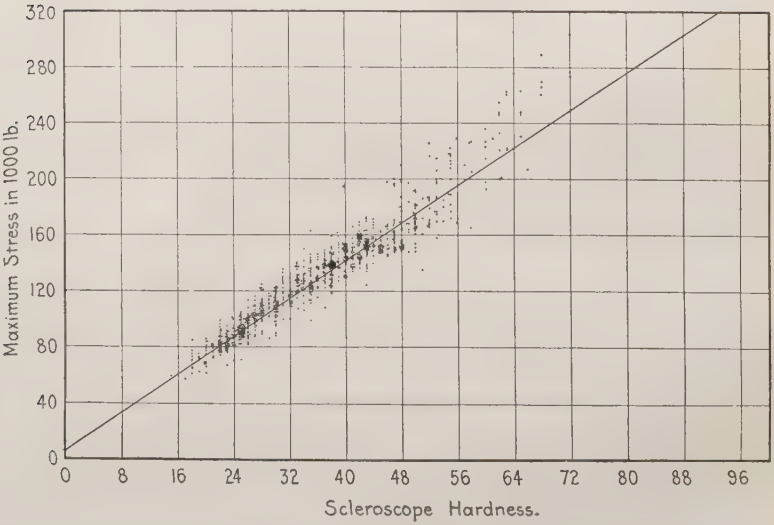


FIG. 5.—Nickel Steels: Relation between Maximum Stress and Scleroscope Hardness.

accurately ground to a finished diameter of 0.505 in., making a cross-sectional area of 0.2 sq. in. Those test bars which were too hard to machine were finished completely by grinding. The test bars were then pulled in a tension test machine, with an autographic recorder. After the test specimens were broken, the long end from each test was machined off, giving a test specimen practically $1\frac{1}{8}$ in. long and $\frac{3}{4}$ in. in diameter. It was believed that by the use of these long ends, any possible discrepancy due to drawing out of the material at the center, during the tension

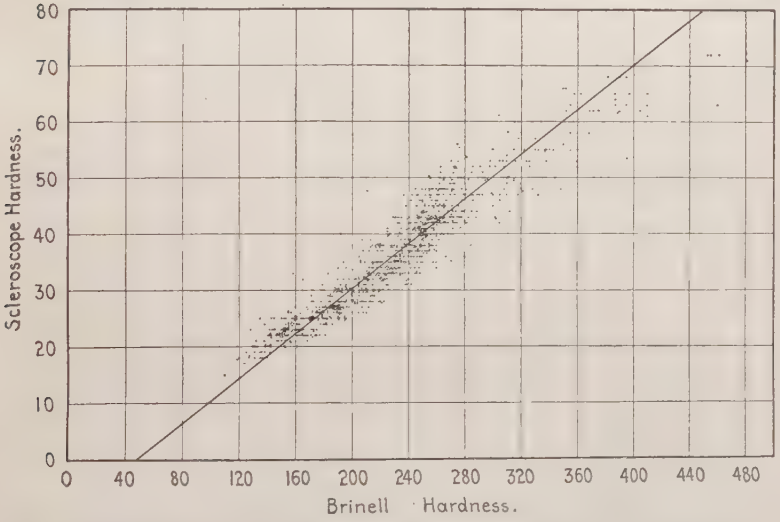


FIG. 6.—Nickel Steels: Relation between Brinell Hardness and Scleroscope Hardness.

test, would be eliminated. Many of the test specimens were cut longitudinally and examined under the microscope for evidence of such drawing out, but none was found. The surface of these small test specimens was then ground on a disk grinder and ten scleroscope tests were made on each piece. The average of these was taken to represent the scleroscope hardness. There was very little difference noted in the hardness at the center, or the outside, and a Brinell test was then made at the center of each specimen. The diameter of the depression was measured under a microscope and the corresponding Brinell number calculated.

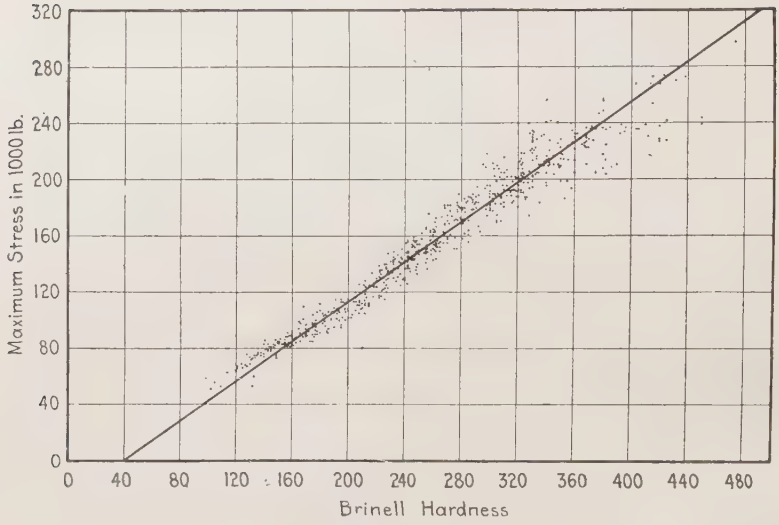


FIG. 7.—Chrome-Vanadium Steels: Relation between Maximum Stress and Brinell Hardness.

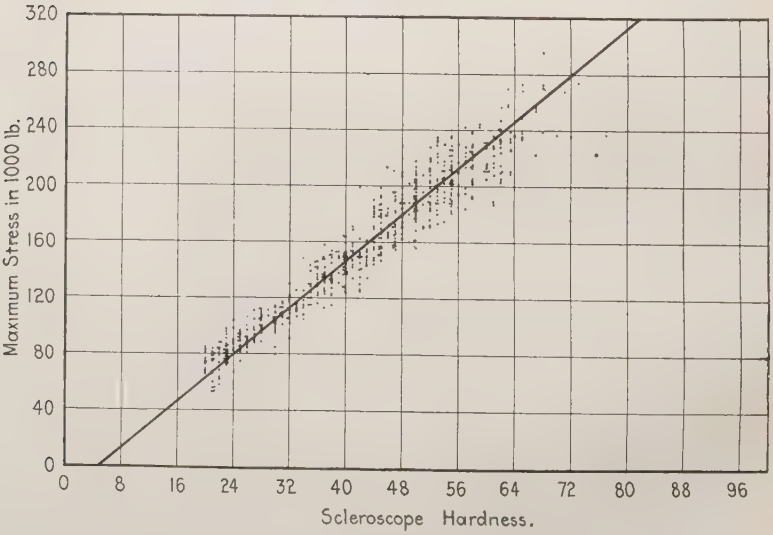


FIG. 8.—Chrome-Vanadium Steels: Relation between Maximum Stress and Scleroscope Hardness.

In the physical tests the elastic limit, maximum strength, rupture stress, reduction of area, elongation, and in addition, the actual foot-pounds of energy necessary to break a specimen, were determined. In this paper only the comparisons are given between the maximum strength and the hardness numbers.

The different steels were then divided into nine groups as follows:

1. Carbon steels;
2. Nickel steels;
3. Chrome-Vanadium steels;

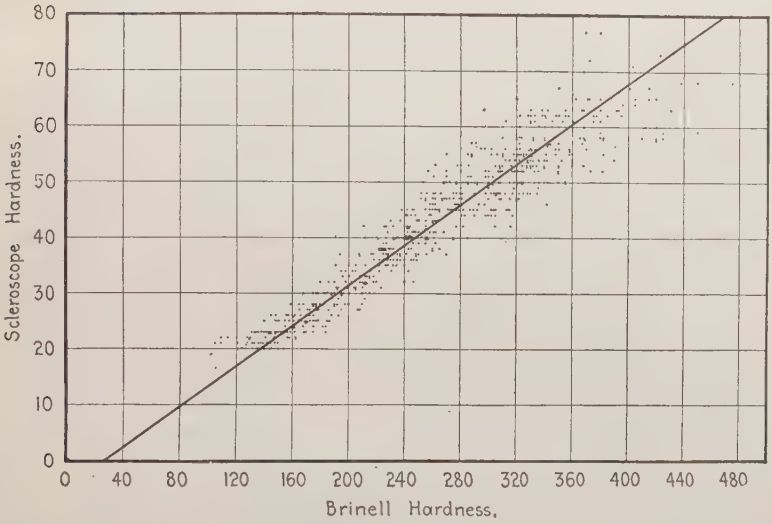


FIG. 9.—Chrome-Vanadium Steels: Relation between Brinell Hardness and Scleroscope Hardness.

4. Chrome-Nickel steels, with nickel content of about 3.5 per cent, and chrome contents about 1 per cent;
5. Chrome-Nickel steels, with nickel contents of about 1.5 per cent, and chrome contents about 0.5 per cent;
6. Silicon steels;
7. Manganese steels;
8. Tungsten steels;
9. Special steels.

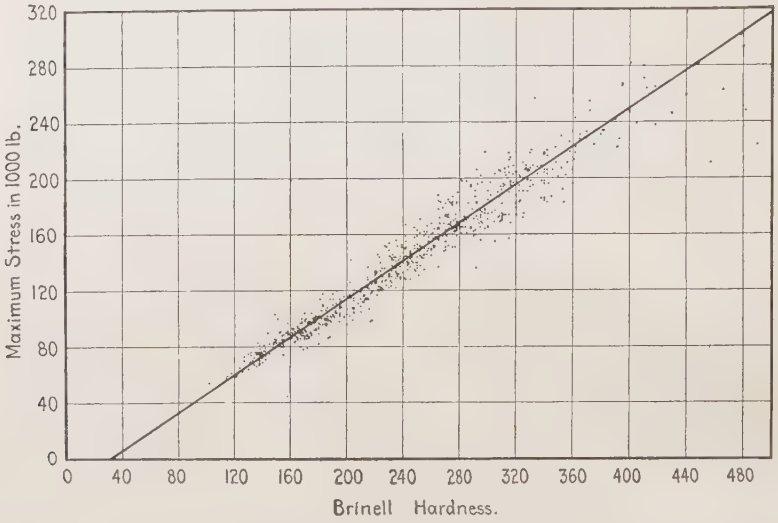


FIG. 10.—Chrome (0.5 per cent) Nickel (1.5 per cent) Steels: Relation between Maximum Stress and Brinell Hardness.

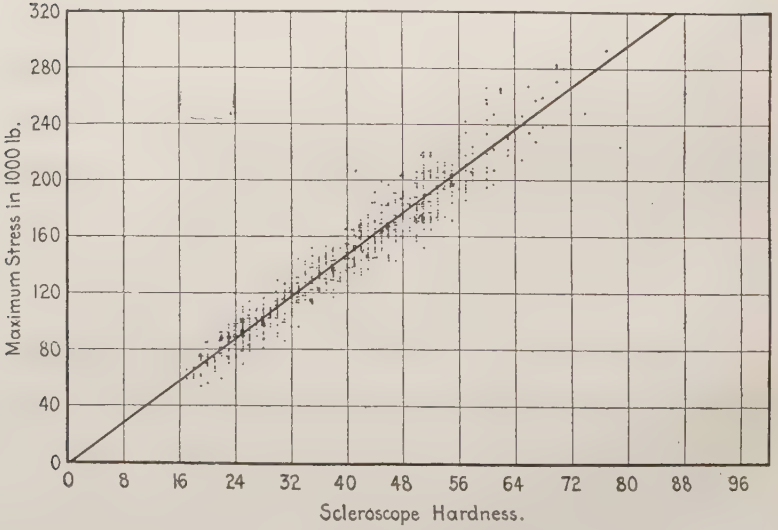


FIG. 11.—Chrome (0.5 per cent) Nickel (1.5 per cent) Steels: Relation between Maximum Stress and Scleroscope Hardness.

In this paper only the first five groups are considered, giving a total of 3932 tests, distributed as follows:

Carbon steel	989
Nickel steel	922
Chrome-vanadium steel	666
High-chrome-nickel steel	628
Low-chrome-nickel steel	727

The relations existing between the three properties, namely, scleroscope hardness, Brinell hardness, and maximum strength,

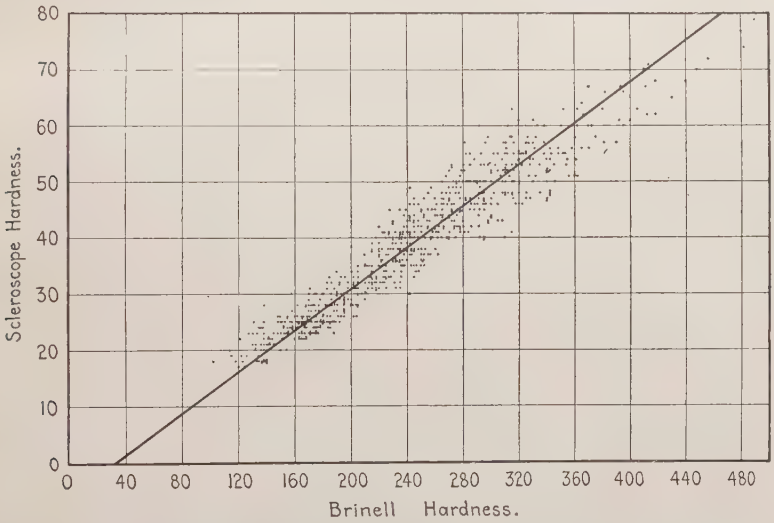


FIG. 12.—Chrome (0.5 per cent) Nickel (1.5 per cent) Steels: Relation between Brinell Hardness and Scleroscope Hardness.

were then plotted upon three different charts for each steel, giving fifteen charts in all. These are shown in Figs. 1 to 15, inclusive, and are self-explanatory, a small dot representing each test. The maximum stress is plotted in units of 1000 lb. per sq. in. In the cases where different tests occupied the same position, separate dots were used for each test, but were necessarily placed a little to the side of their true position.

From a consideration of the results shown in Figs. 1 to 15, inclusive, it is evident that within reasonable limits they all lie approximately along a straight line, or, expressed mathematically,

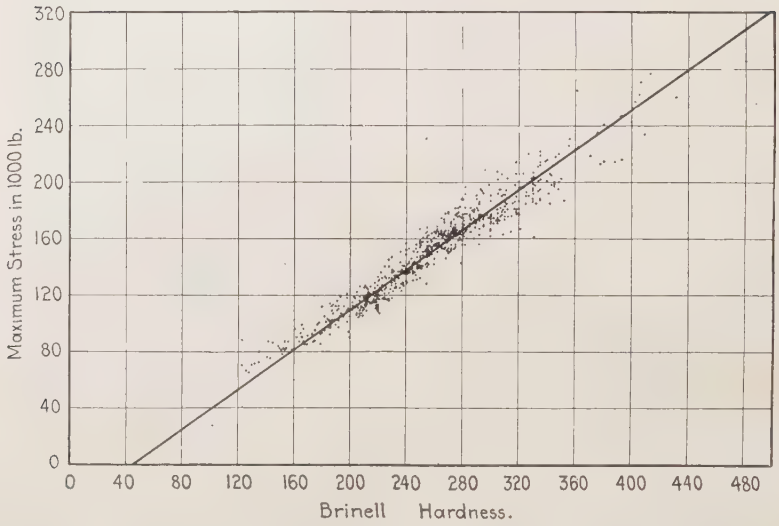


FIG. 13.—Chrome (1.0 per cent) Nickel (3.5 per cent) Steels: Relation between Maximum Stress and Brinell Hardness.

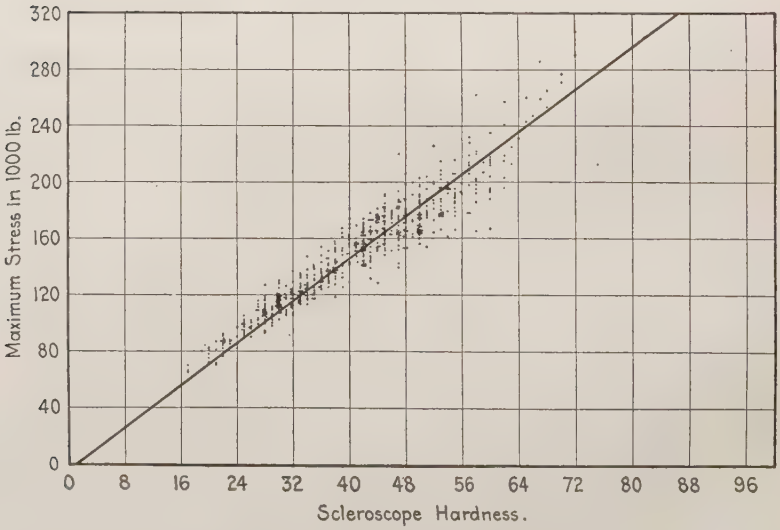


FIG. 14.—Chrome (1.0 per cent) Nickel (3.5 per cent) Steels: Relation between Maximum Stress and Scleroscope Hardness.

the relations existing between Brinell and maximum, scleroscope and maximum, and Brinell and scleroscope, are straight-line functions of each other. It will be noted on all the Brinell curves that there is a tendency to a curvature to the left at the lower end. This is particularly noticeable in Fig. 1. At first it was believed that these functions were not straight lines, but it was finally found that none of the ordinary curves would approximate this as closely as a straight line. It has now become apparent that on steels with a maximum strength below 55,000 lb.

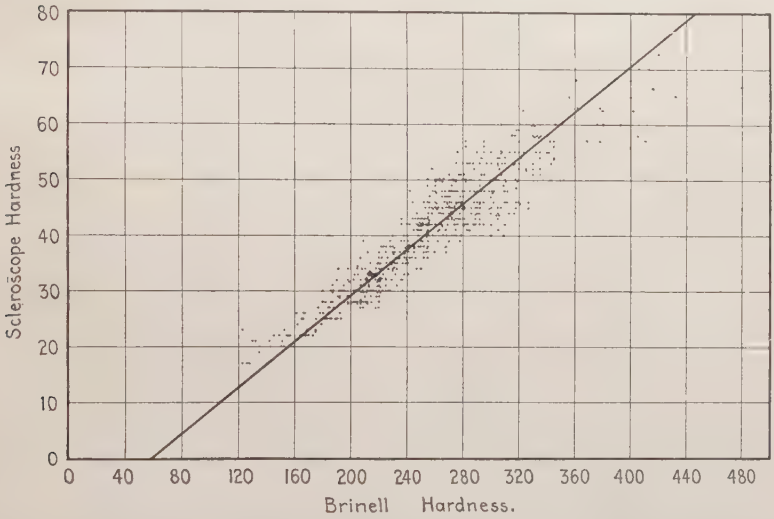


FIG. 15.—Chrome (1.0 per cent) Nickel (3.5 per cent) Steels: Relation between Brinell Hardness and Scleroscope Hardness.

per sq. in. or a Brinell number of less than 110, the size of test specimen used ($\frac{3}{4}$ in. in diameter) was not quite large enough to prevent a slightly greater Brinell depth than would have been the case with a larger specimen.

After plotting the positions of the various points on the curves, it becomes desirable to locate a line which will most nearly approximate the average position of these points. The ordinary method of doing this is to lay out this line by the eye, endeavoring to place it upon the average central position of the group of points. A better method would be to determine the

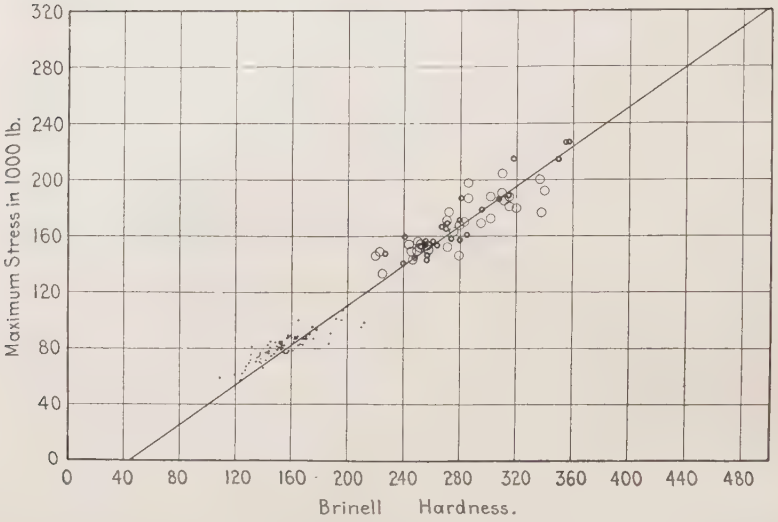


FIG. 16.—Nickel Steels: Relation between Maximum Stress and Brinell Hardness.

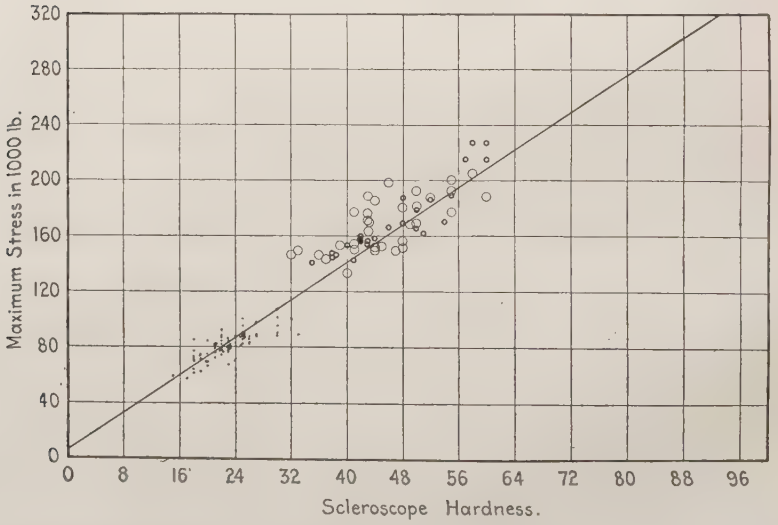


FIG. 17.—Nickel Steels: Relation between Maximum Stress and Scleroscope Hardness.

line mathematically. The method of least squares will locate its most probable position. For the location of the lines shown in Figs. 1 to 15, this method has been used in calculating their equations, and the lines have then been plotted from the equations. It will be seen that the lines as plotted represent the average locus of the points very well.

This method of locating the probable equation of a group of points can be likened to the following problem: Suppose a town to contain 1000 houses, and to make the problem more

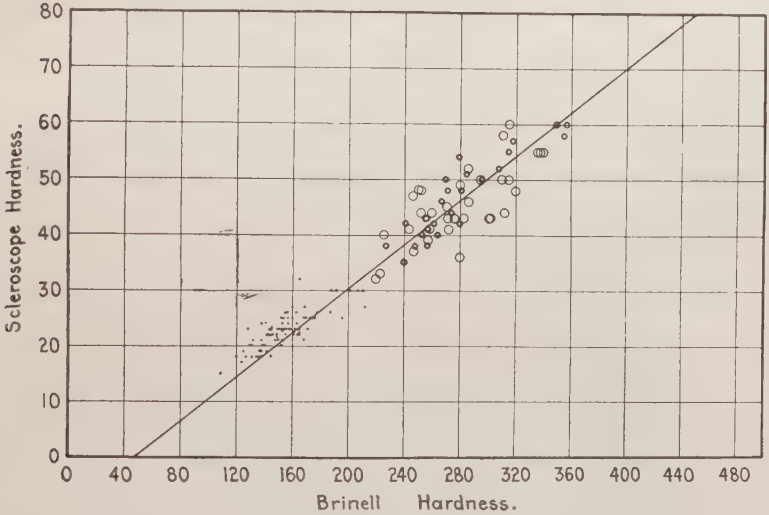


FIG. 18.—Nickel Steels: Relation between Brinell Hardness and Scleroscope Hardness.

similar, imagine these houses to be strung out along an approximately straight line. It is desired to light each house by electricity, and in order to do this a main cable is to be run in a straight line through this town and feeder wires are to be run from this cable to each house. Practically, then, the problem would be to so locate the main cable that the sum of the lengths of the separate wires to each house should be a minimum. The method of least squares will readily approximate this line.

The equations of the curves are given in Tables I, II and III.

TABLE I.—EQUATIONS CONNECTING MAXIMUM STRENGTH AND BRINELL NUMBER.

Kind of Steel.	Equation.
Carbon steel	$M = 0.73\,B - 28$
Nickel steel	$M = 0.71\,B - 32$
Chrome-vanadium steel	$M = 0.71\,B - 29$
Low-chrome-nickel steel	$M = 0.68\,B - 22$
High-chrome-nickel steel	$M = 0.71\,B - 33$
All steels grouped together	$M = 0.70\,B - 26$

TABLE II.—EQUATIONS CONNECTING MAXIMUM STRENGTH AND SCLEROSCOPE NUMBER.

Kind of Steel.	Equation.
Carbon steel	$M = 4.4\,S - 28$
Nickel steel	$M = 3.5\,S - 6$
Chrome-vanadium steel	$M = 4.2\,S - 21$
Low-chrome-nickel steel	$M = 3.7\,S - 1$
High-chrome-nickel steel	$M = 3.7\,S - 3$
All steels grouped together	$M = 4.0\,S - 15$

TABLE III.—EQUATIONS CONNECTING BRINELL AND SCLEROSCOPE NUMBERS.

Kind of Steel.	Equation.
Carbon steel	$B = 5.6\,S + 14$
Nickel steel	$B = 5.0\,S + 48$
Chrome-vanadium steel	$B = 5.5\,S + 27$
Low-chrome-nickel steel	$B = 5.4\,S + 33$
High-chrome-nickel steel	$B = 4.8\,S + 58$
All steels grouped together	$B = 5.5\,S + 28$

TABLE IV.—MAXIMUM STRENGTH CORRESPONDING TO DIFFERENT BRINELL VALUES AS DETERMINED BY THE EQUATIONS OF TABLE I.

Brinell.	Maximum Strength, lb. per sq. in.					
	Carbon Steel.	Nickel Steel.	Chrome-Vanadium Steel.	Low-Chrome-Nickel Steel.	High-Chrome-Nickel Steel.	All Steels.
100	45 000	39 000	42 000	46 000	38 000	44 000
150	81 000	74 000	77 000	80 000	73 000	79 000
200	118 000	110 000	113 000	114 000	109 000	114 000
250	154 000	145 000	148 000	148 000	144 000	149 000
300	191 000	181 000	184 000	182 000	180 000	184 000
350	227 000	216 000	219 000	216 000	215 000	219 000
400	264 000	252 000	255 000	250 000	251 000	254 000
450	300 000	287 000	290 000	284 000	286 000	289 000
500	337 000	323 000	326 000	318 000	322 000	324 000
550	373 000	358 000	361 000	352 000	357 000	359 000
600	410 000	394 000	397 000	386 000	393 000	394 000

TABLE V.—MAXIMUM STRENGTH CORRESPONDING TO DIFFERENT SCLEROSCOPE VALUES AS DETERMINED BY THE EQUATIONS OF TABLE II.

Scleroscope.	Maximum Strength, lb. per sq. in.					
	Carbon Steel.	Nickel Steel.	Chrome-Vanadium Steel.	Low-Chrome-Nickel Steel.	High-Chrome-Nickel Steel.	All Steels.
20	60 000	64 000	63 000	73 000	71 000	65 000
30	104 000	99 000	105 000	110 000	108 000	105 000
40	148 000	134 000	147 000	147 000	145 000	145 000
50	192 000	169 000	189 000	184 000	182 000	185 000
60	236 000	204 000	231 000	221 000	219 000	225 000
70	280 000	239 000	273 000	258 000	256 000	265 000
80	324 000	274 000	315 000	295 000	293 000	305 000
90	368 000	309 000	357 000	332 000	330 000	345 000
100	412 000	344 000	399 000	369 000	367 000	385 000

In Tables I, II, III, M = the maximum strength in units of 1000 lb. per sq. in.; B = the Brinell hardness number; S = the scleroscope hardness number.

It will be noticed that in none of the equations do the lines pass through the origin. That is to say, in no case can the relation between the two factors be expressed by a single number. The nearest approach to this is in the equation connecting maximum strength and scleroscope number. Tables IV, V and VI represent the values obtained by substituting in the equation of Tables I, II and III.

TABLE VI.—BRINELL HARDNESS NUMBER CORRESPONDING TO DIFFERENT SCLEROSCOPE VALUES AS DETERMINED BY EQUATIONS OF TABLE III.

Scleroscope.	Brinell Hardness Number.					
	Carbon Steel.	Nickel Steel.	Chrome-Vanadium Steel.	Low-Chrome-Nickel Steel.	High-Chrome-Nickel Steel.	All Steels.
20	126	148	137	141	154	138
30	182	198	192	195	202	193
40	238	248	247	249	250	248
50	294	298	302	303	298	303
60	350	348	357	357	346	358
70	406	398	412	411	394	413
80	462	448	467	465	442	468
90	518	498	522	519	490	523
100	574	548	577	573	538	578

It was next thought desirable to determine more closely the accuracy of the equations of Tables I, II and III, when applied to annealed and heat-treated steels. The results of the investigation for nickel steels only are given in Figs. 16, 17 and 18. The equations represented by the formulas for nickel steels of Tables I, II and III are reproduced; the dots represent perfectly annealed steels. In Figs. 16 and 17 these lie slightly to the left of the line. This is due, as explained before, to the softer steels giving a lower Brinell reading on account of the small diameter of the test specimen. A further confirmation of this lies in the fact that on Fig. 17, showing the relation between

maximum and scleroscope, these dots agree much better with the line.

The next step was to investigate the difference in the relations of the three factors, Brinell number, scleroscope number and maximum strength on two steels, one with a low, the other with a higher carbon content, but heat-treated to bring out practically the same maximum strength. The results for nickel steels are here reproduced. Two groups of steels were selected, one with carbon below 0.20 per cent and the other above 0.35 per cent. Tests of heat-treated steels showing maximum strength of from 125,000 to about 225,000 lb. per sq. in. were then selected from both groups and plotted in Figs. 16, 17 and 18. The small circles represent the high and the large circles the low-carbon steels. A broad consideration of Figs. 16, 17 and 18 shows very little difference and we can therefore conclude that if a low and high-carbon steel are heat-treated to the same strength, the hardness will be the same, and also that the relation between the Brinell and scleroscope readings will correspond.

There is noticed a tendency towards a greater variation from the line of the equation with the low than with the high-carbon steels.

DISCUSSION.

Mr. Zimmerschied.

MR. K. W. ZIMMERSCHIED.—We are very much indebted to Mr. Abbott for furnishing such strong evidence that the Brinell and scleroscope hardness are functions of the maximum strength and not of chemical composition or heat treatment. We are sorry, however, to be assured that these tests do not bear any generic relation to the elastic limit, as the latter property is the one on which most engineers must base their final calculations.

Of course, for a given type of steel and heat treatment, it is possible to establish an empirical relation which will hold within a given range of commercial limits, but most engineers who are concerned with highly stressed parts and who are confined to very limited factors of safety, would be grateful for such simple tests as these, if they would show a definite relation to the elastic limit.

Mr. Wille.

MR. H. V. WILLE.—I have had very little experience with Brinell's method of testing for hardness except on one grade of steel, and that is tire steel. For a time we endeavored to apply a modified form of Brinell's test to every heat-treated tire. The steel had a tensile strength corresponding to carbon between 0.60 and 0.70 per cent, and I looked up some of these results within the last few days. For that grade of steel I can say that the results shown by Mr. Abbott are about the same as we obtained on a test of full-size tires. The machine on which the sample was tested was not a regular Brinell machine, but was a large machine built by Olsen for the purpose of testing full-size specimens, and even those tests f full-size specimens confirm, for that particular grade of steel, the results given by Mr. Abbott.

Mr. Abbott.

MR. ROBERT R. ABBOTT.—In reply to Mr. Zimmerschied, it would be very desirable if we could correlate the elastic limit with Brinell or scleroscope hardness—however, that seems almost impossible. We can console ourselves, however, with the fact,

that in most heat-treated steel the elastic limit is brought decidedly nearer the maximum strength. Particularly in heat-treated steels of high strength, such as are now being used very extensively, the elastic limit is so close that, knowing the steel, we can almost figure how much lower the elastic limit is than the maximum strength, so we can at least make an intelligent guess. Mr. Abbott.

THE CHARPY IMPACT TEST ON HEAT-TREATED STEELS.

By J. J. Thomas.

SUMMARY

This paper describes the Charpy impact machine and then gives in detail four experiments made with it. The first was upon a piece of heat-treated steel taken from a broken gun jacket. By reheating, the impact strength of this steel was increased three-fold, although the elastic limit and tensile strength were not changed.

The second experiment was made with nickel-vanadium steel, in which forged and cast metals were given various heat treatments, and the physical qualities compared. The forged-steel specimens drawn at a temperature slightly below Ac1 gave the greatest resistance to shock. For similar heat treatments the forged steel was tougher and more ductile than the cast.

The third experiment gives the results obtained with a low-carbon steel quenched and drawn at various temperatures below Ac1. The rate of cooling after drawing was varied by cooling in water, air and furnace. The water-cooled specimens were harder and more brittle than those cooled in the furnace, although the drawing temperatures were below Ac1.

The fourth experiment determines the effect of the shape of the notch in the Charpy test bar. Flaring the sides of the notch to an angle of 30 deg. from the normal apparently has no effect.

The Charpy impact test appears to differentiate between steels of various degrees of brittleness where the ordinary tension test often fails to do so. Comparison should never be made, however, between single specimens. An average of four or more tests should always be made.

THE CHARPY IMPACT TEST ON HEAT-TREATED STEELS.

By J. J. Thomas.

Description of Machine.

The object of this paper is to describe a number of experiments made with the Charpy impact machine, for the purpose

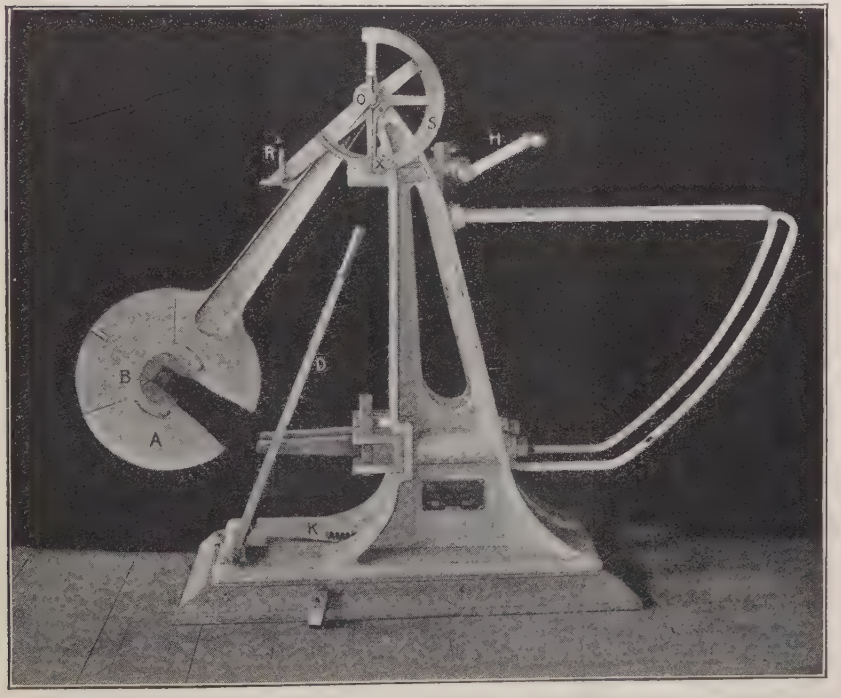


Fig. 1.—Charpy Impact Machine.

of determining how the shock-resisting qualities of various steels are affected by heat treatment.

Figs. 1 and 2 illustrate the type of machine used. The mounted pendulum *A* swings about the point *O*, and is capable

of delivering a blow having a total energy of 30 m-kg. (216.99 ft-lb.). *B* is a hardened steel knife edge rounded to a radius of 2 mm. The machine is operated by raising the pendulum to an angle of 155 deg. from its lower vertical position, by means of the handle *H* and connecting worm and wheel. The pointer at *X* is then set to read zero on the graduated scale *S*.

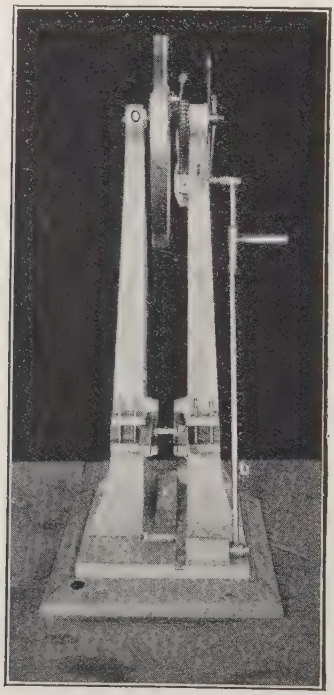


FIG. 2.—Charpy Impact Machine.

The test specimen, a small bar 10 by 10 by 60 mm., notched to a depth of 5 mm. at the center, the bottom of notch having a radius of 0.667 mm., is then placed across the knife edges at *E* (see Fig. 3), and the pendulum released by the trigger *R*. The test specimen is literally chopped in two, after which the pendulum ascends to an angle depending on the energy remaining after impact. This angle is registered by the needle on the scale *S*.

Knowing the weight of the pendulum and the height of its center of gravity before impact, the energy of the blow can be determined. The height of the center of gravity, for the angle of ascension after impact, is registered on the scale *S*, and thus the energy remaining after impact can be determined. The difference is, of course, the energy absorbed by the test specimen, and is the value reported for the test. The lever *D* operates a brake *K*, which brings the pendulum to rest after the test.

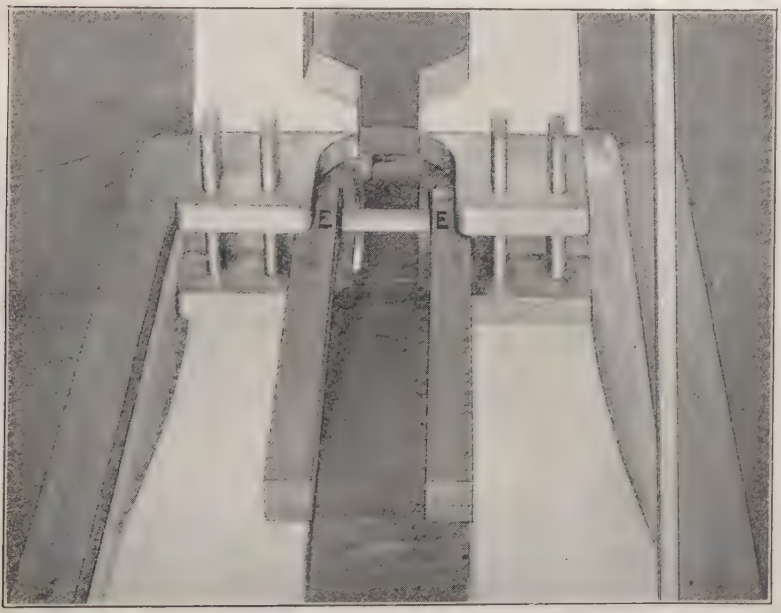


FIG. 3.—Test Specimen in Place in the Machine.

Fig. 3 illustrates how the test specimen is placed in the machine, the slot always being on the side opposite the point of impact.

TEST OF BROKEN GUN JACKET.

A fragment of a broken gun jacket was taken and the metal tested in the original condition; that is, after it had been heat-treated as a large gun forging. Other pieces were tested after

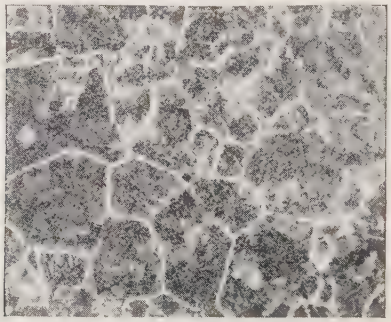


FIG. 4.—Specimen No. 1; Metal Tested in Original Condition.

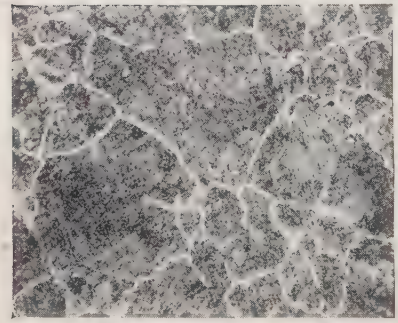


FIG. 5.—Specimen No. 2; Reheated to 500° C.

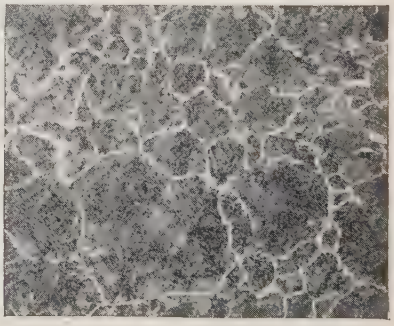


FIG. 6.—Specimen No. 3; Reheated to 600° C.

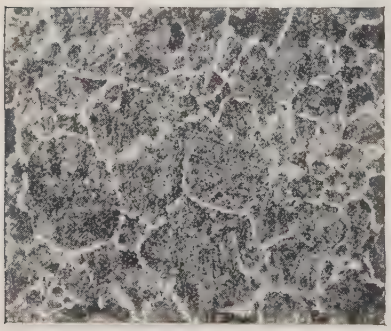


FIG. 7.—Specimen No. 4; Reheated to 700° C.

FIG. 8.—Specimen No. 5; Reheated to 750° C.

MICROPHOTOGRAPHS OF A FRAGMENT OF A BROKEN GUN JACKET. MAGNIFICATION, × 45.

reheating to 500, 600, 700 and 750° C., respectively, for half an hour, cooling in the furnace. Tension, Charpy impact and hardness tests were made, and microphotographs were taken as shown in Figs. 4 to 8, inclusive.

From the results of the Charpy tests given in Table I it will be noted that there is considerable variation between the strengths of individual pieces. For this reason, the average of

TABLE I.—TESTS OF BROKEN GUN JACKET.

Specimen No.	Heat Treatment.	Charpy Impact, m-kg.	Elastic Limit, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent.	Reduction of Area, per cent.	Brinell Hardness Number.
1		51.5					
		50.6	50 000	101 000	20.0	27.4	187
		26.2					
		64.1					
Average...		48.1					
2	Reheated to 500° C.	134.0					
		178.2	50 000	100 500	20.0	37.2	187
		147.8					
		154.8					
Average...		154.0					
3	Reheated to 600° C.	140.8					
		161.7	49 000	99 500	21.0	40.3	187
		162.0					
		139.0					
Average...		151.0					
4	Reheated to 700° C.	118.8					
		148.1	45 500	90 500	25.0	49.1	162
		148.1					
		173.0					
Average...		147.0					
5	Reheated to 750° C.	147.9					
		133.4	47 000	91 500	22.5	34.0	159
		195.6					
		151.0					
		158.0					

four or more tests should always be taken, before reliable comparisons can be made.

It is interesting to note that for the specimens heated to 600 and 500° C., the elastic limit, tensile strength, elongation and Brinell hardness are the same as for the original steel, yet the resistance to shock has increased three-fold. The reduction of area has increased, and this value alone indicates the changed condition of the metal. Figs. 4, 5, 6 and 7, illustrating the structure of test specimens Nos. 1, 2, 3 and 4, do not differ

materially in grain size, but this could hardly be expected as the metal was not heated above the Ac1 point (739° C.). For specimen No. 5 the metal was heated above Ac1 and there has been a distinct refining of the grain (see Fig. 8), and a decrease of hardness and strength. From these results it is seen that brittleness is not due to grain size alone. It is probable that the weak impact strength of the original metal was due to internal strains which were relieved by the subsequent reheating.

TESTS OF CAST AND FORGED ALLOY STEELS.

For this experiment an ingot of nickel-vanadium steel of the following composition was taken:

Carbon	0.455 per cent.
Manganese	0.50 "
Silicon	0.23 "
Sulfur	0.049 "
Phosphorus	0.033 "
Nickel	3.45 "
Vanadium	0.27 "

The ingot was cast in a dry-sand mold 20 in. in diameter and 50 in. long. After casting, the ingot was heated to 950° C., air-chilled, and then returned to the furnace at 400° C. and allowed to cool with the furnace. It was again heated to 1000° C. and air-chilled. A slab 4 in. thick and 20 in. diameter was then cut from the ingot and split across a diameter. One-half was forged down to a slab 5 in. wide by 1 in. thick, the other half remaining in the cast condition. The forging operations were all normal and the forging finished above the Ac1 point (710° C.).

Specimens of both cast and forged steels were tested in the original condition, after which the remaining pieces were given the heat treatments shown in Table II. Both cast and forged steels were given exactly the same heat treatments for the same length of time, in order that a direct comparison could be made. The curves in Figs. 9 and 10 show graphically the results obtained.

With the exception of the two lower drawing temperatures, 350 and 450° C., we see that the same heat treatment has produced practically the same elastic limit and tensile strength, but that the resistance to shock and the ductility have been

TABLE II.—TESTS OF CAST AND FORGED ALLOY STEELS.

CAST STEEL.

Specimen No.	Heat Treatment.	Elastic Limit, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent.	Reduction of Area, per cent.	Charpy Impact, ft-lb. per sq. in.	Brinell Hardness Number.
3150AX	As cast	73 500	93 000	2.0	5.7	Maximum, 24.3 Minimum, 11.4 Average,[a] 15.5	Maximum, 248 Minimum, 228 Average, 242
3150A6[b]	Oil quenched from 770° C.; drawn one-half hour at 710° C.	71 000	99 500	11.0	13.3	Maximum, 186.0 Minimum, 178.7 Average,[a] 182.3	Maximum, 207 Minimum, 202 Average, 205
3150A1[b]	Oil quenched from 770° C.; drawn one-half hour at 700° C.	79 500	99 500	13.0	13.3	Maximum, 194.0 Minimum, 163.5 Average,[a] 176.0	Maximum, 212 Minimum, 196 Average, 200
3150A2[b]	Oil quenched from 770° C.; drawn one-half hour at 650° C.	117 000	128 500	10.5	24.0	Maximum, 178.7 Minimum, 178.2 Average,[a] 178.5	Maximum, 228 Minimum, 223 Average, 225
3150A3[b]	Oil quenched from 770° C.; drawn one-half hour at 550° C.	141 500	148 000	3.0	5.7	Maximum, 189.7 Minimum, 64.6 Average,[a] 127.4	Maximum, 321 Minimum, 311 Average, 316
3150A4[b]	Oil quenched from 770° C.; drawn one-half hour at 450° C.	162 000	171 500	5.5	13.3	Maximum, 117.5 Minimum, 64.6 Average,[a] 77.8	Maximum, 364 Minimum, 364 Average, 364
3150A5[b]	Oil quenched from 770° C.; drawn one-half hour at 350° C.	163 000	202 500	1.5	5.7	Maximum, 39.1 Minimum, 23.0 Average,[a] 28.3	Maximum, 444 Minimum, 444 Average, 444

FORGED STEEL.

3150BX	As forged	96 500	144 000	12.5	20.5	Maximum, 91.6 Minimum, 73.7 Average,[a] 83.8	Maximum, 286 Minimum, 277 Average, 284
3150B6[b]	Oil quenched from 770° C.; drawn one-half hour at 710° C.	75 000	101 500	24.0	51.9	Maximum, 231.0 Minimum, 214.0 Average,[a] 226.0	Maximum, 196 Minimum, 187 Average, 193
3150B1[b]	Oil quenched from 770° C.; drawn one-half hour at 700° C.	81 000	105 500	22.5	46.2	Maximum, 320.5 Minimum, 231.4 Average,[a] 266.8	Maximum, 228 Minimum, 196 Average, 211
3150B2[b]	Oil quenched from 770° C.; drawn one-half hour at 650° C.	107 500	121 500	19.0	49.1	Maximum, 320.5 Minimum, 155.1 Average,[a] 246.8	Maximum, 302 Minimum, 241 Average, 264
3150B3[b]	Oil quenched from 770° C.; drawn one-half hour at 550° C.	143 500	147 500	15.1	40.3	Maximum, 201.2 Minimum, 150.0 Average,[a] 176.1	Maximum, 321 Minimum, 269 Average, 300
3150B4[b]	Oil quenched from 770° C.; drawn one-half hour at 450° C.	151 500	157 000	12.0	34.0	Maximum, 166.0 Minimum, 132.0 Average,[a] 144.1	Maximum, 340 Minimum, 321 Average, 326
3150B5[b]	Oil quenched from 770° C.; drawn one-half hour at 350° C.	180 500	191 500	7.5	20.5	Maximum, 120.5 Minimum, 64.7 Average,[a] 88.6	Maximum, 418 Minimum, 387 Average, 392

[a] Six specimens tested.

[b] All specimens heated for 1½ hours and air-chilled from 1000° C. before oil-quenching as shown.

greatly increased by forging. The maximum increase in impact strength appears to be reached in both cases at a drawing tem-

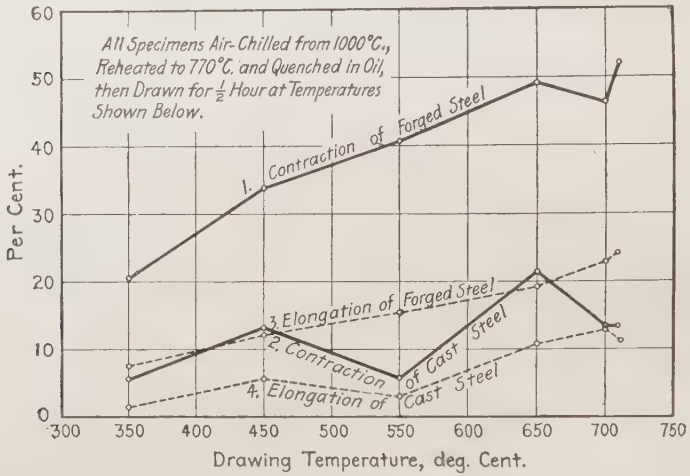

All Specimens Air-Chilled from 1000°C., Reheated to 770°C. and Quenched in Oil, then Drawn for ½ Hour at Temperatures Shown Below.
1. Contraction of Forged Steel
3. Elongation of Forged Steel
2. Contraction of Cast Steel
4. Elongation of Cast Steel
Per Cent.
Drawing Temperature, deg. Cent.

FIG. 9.—Effect of Drawing Temperature on Elongation and Contraction.

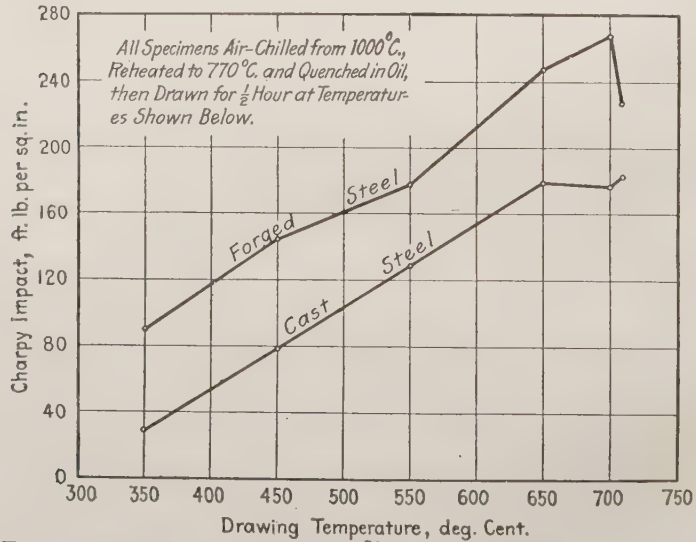

All Specimens Air-Chilled from 1000°C., Reheated to 770°C. and Quenched in Oil, then Drawn for ½ Hour at Temperatures Shown Below.
Forged Steel
Cast Steel
Charpy Impact, ft. lb. per sq. in.
Drawing Temperature, deg. Cent.

FIG. 10.—Effect of Drawing Temperature on Resistance to Shock.

perature of 650° C. Higher drawing temperatures produce no material increase in shock strength, probably due to the fact

that the steel is entering the critical range and a transformation, or recrystallization, occurs. A heating-cooling curve for this steel is shown in Fig. 11.

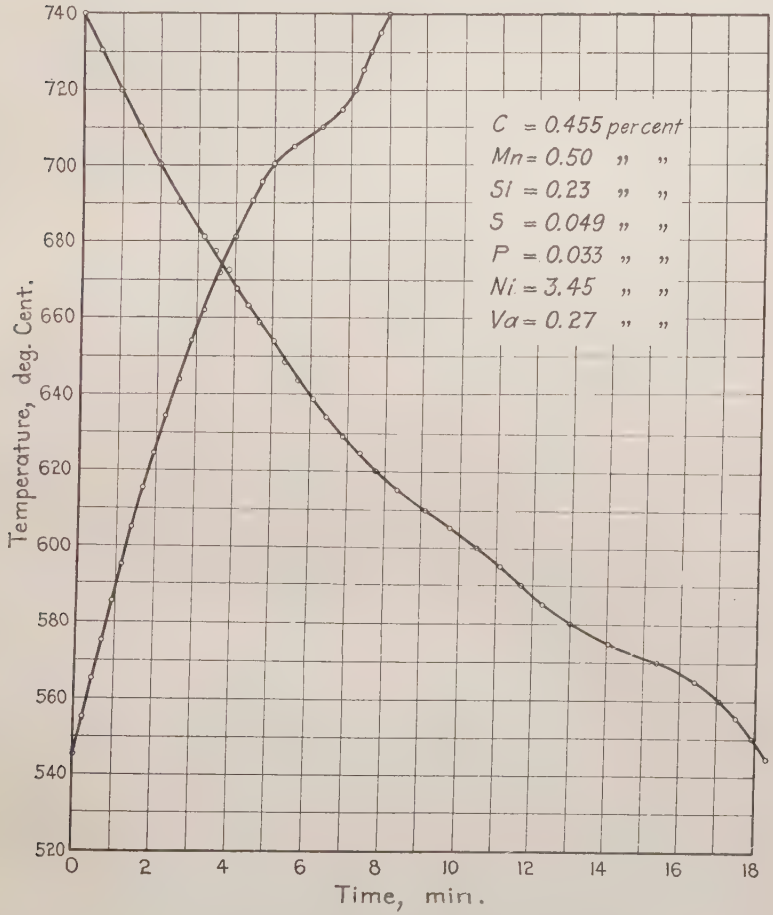


FIG. 11.—Heating-Cooling Curves for Nickel-Vanadium Steel.

EFFECT OF RATE OF COOLING AFTER DRAWING (OR TEMPERING).

The general plan for carrying out this investigation was as follows: Twenty-four similar pieces of a hypoeutectoid steel,

all of which should be in the same physical condition and each large enough to cut into six Charpy test specimens, were to be quenched in water from 25° C. above the Ar3 point. These 24 pieces of hardened steel were to be drawn as follows:

Draw 3 at 300° C.	Cool one in furnace	Mark S3F
	" " " air	" S3A
	" " " water	" S3W
Draw 3 at 400° C.	" " " furnace	" S4F
	" " " air	" S4A
	" " " water	" S4W
Draw 3 at 500° C.	" " " furnace	" S5F
	" " " air	" S5A
	" " " water	" S5W
Draw 3 at 600°C.	" " " furnace	" S6F
	" " " air	" S6A
	" " " water	" S6W
Draw 3 at 650° C.	" " " furnace	" S65F
	" " " air	" S65A
	" " " water	" S65W
Draw 3 at 675° C.	" " " furnace	" S67F
	" " " air	" S67A
	" " " water	" S67W
Draw 3 at 700° C.	" " " furnace	" S7F
	" " " air	" S7A
	" " " water	" S7W
Draw 3 at 725° C.	" " " furnace	" SCF
	" " " air	" SCA
	" " " water	" SCW

For this experiment a mild steel of the following composition was chosen:

Carbon	0.20	per cent
Manganese	0.40	"
Silicon	0.032	"
Sulfur	0.033	"
Phosphorus	0 012	"

All heating operations were carried out in a small electric muffle furnace. The temperatures were measured by a calibrated pt. pt-rh. couple used with a Leeds and Northrup precision potentiometer. All pieces were heated to 900° C., allowed to cool to 825° C., and then quenched in cold water. The specimens were then placed in the furnace, brought to the desired drawing temperature, held one-half hour and cooled in furnace, air or water, as indicated in the program.

TABLE III.—EFFECT OF RATE OF COOLING SPECIMEN.

Mark.	Charpy Impact, ft-lb. per sq. in.			Percentage Lower than Furnace-Cooled.	Brinell Hardness Number.	Method of Drawing.
	Maximum.	Minimum.	Average of Six Specimens.			
S3F	631.5	418.0	531.6	0.0	170.3	Furnace-cooled from 300° C.
S3A	609.0	358.0	536.6	0.9[a]	200.0	Air " " "
S3W	568.0	323.0	452.3	14.9	207.3	Water " " "
S4F	632.0	400.0	511.2	0.0	183.3	Furnace " " 400° C.
S4A	609.0	388.0	531.0	3.9[a]	192.7	Air " " "
S4W	459.0	370.0	397.2	22.3	224.7	Water " " "
S5F	600.0	360.5	488.9	0.0	171.3	Furnace " " 500° C.
S5A	566.0	341.0	434.4	11.1	183.3	Air " " "
S5W	576.0	360.5	467.5	4.4	177.3	Water " " "
S6F	643.5	351.0	514.3	0.0	154.7	Furnace " " 600° C.
S6A	666.0	360.5	541.4	5.3[a]	145.0	Air " " "
S6W	726.0	268.0	464.3	9.7	164.7	Water " " "
S65F	760.0	406.5	606.5	0.0	143.2	Furnace " " 650° C.
S65A	680.0	339.0	534.0	12.0	145.0	Air " " "
S65W	658.0	360.5	471.25	22.3	160.0	Water " " "
S67F	726.0	410.0	577.75	0.0	140.5	Furnace " " 675° C.
S67A	678.0	360.5	544.75	5.7	154.0	Air " " "
S67W	678.0	390.0	516.2	10.7	146.0	Water " " "
S7F	780.0	460.0	605.7	0.0	129.4	Furnace " " 700° C.
S7A	680.0	341.5	566.75	6.4	129.5	Air " " "
S7W	646.0	303.0	484.0	20.1	143.0	Water " " "
SCF	330.0	258.0	299.75	0.0	114.2	Furnace " " 725° C.
SCA	481.5	312.0	358.9	19.7[a]	118.2	Air " " "
SCW	547.0	398.0	475.0	58.5[a]	164.0	Water " " "

[a] Increased percentages.

Standard Charpy test specimens were machined from the heat-treated pieces and tested as indicated in Table III. The averages given are those of 6 tests in every case.

As with the tests of alloy steels it will be noted that the maximum resistance to shock is obtained by drawing at 650° C. An examination of Table III reveals the fact that in every case the average resistance to shock is less for the water-cooled specimens than for those cooled in the furnace. This decrease

in the resistance to shock varies from 4 to 22 per cent. The air-cooled pieces, although more inconsistent, show a similar tendency. For the three upper drawing temperatures, the resistance to shock of the air-cooled specimens lies between the furnace and water-cooled specimens, as might be expected.

In the case of the specimens drawn at 725° C., the water-cooled show the greatest resistance to shock, the air-cooled next, and the furnace-cooled least. This is explained by the fact that the specimens having been heated to 725° C., had passed the Ac1 point, and in the case of the slowly cooled specimens a true annealing had occurred. The specimens which were water-

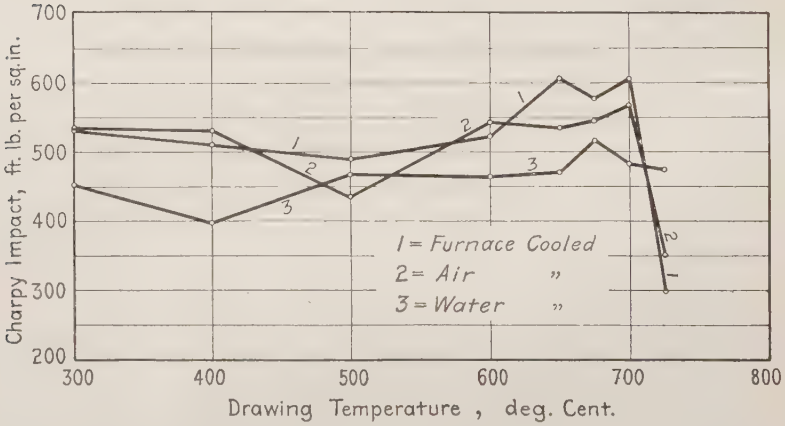


FIG. 12.—Effect of Rate of Cooling after Drawing on Resistance to Shock.

quenched at this temperature were hardened in the critical range and gave a shock test almost equal to the specimens which were drawn at 700° C. The results are shown graphically in Figs. 12 and 13.

It will also be noted in Table III that the Brinell hardness is greater in every case for the water-cooled than for the furnace-cooled specimens, in spite of the fact that all specimens were quenched below the Ac1 point where no critical points exist, and no allotropic changes occur. It appears to the author that as these variations in hardness occur below Ac1 they must be independent of the carbon content of the steel, and therefore, the results

obtained with this 0.20-per-cent carbon-steel would hold good for steels of varying carbon content. It is probable that the variations in hardness and toughness may be due to internal stresses set up, caused by unequal contraction in the material during quenching. A discussion of this point is invited.

EFFECT OF THE SHAPE OF THE NOTCH.

With a view to determining the effect of the shape of the notch on the results of Charpy impact tests, the following experiment was carried out:

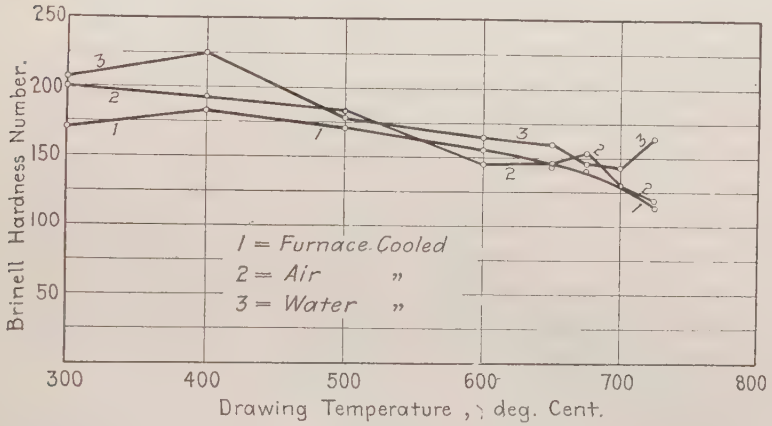


FIG. 13.—Effect of Rate of Cooling After Drawing on Resistance to Shock.

The Charpy impact machine used is one of 30 m-kg. or 216.99 ft-lb. capacity. The standard transverse specimen for this machine is 10 by 10 by 60 mm. with a notch 1.3 mm. wide by 5 mm. deep, midway along one side. This is a difficult piece to machine from hard steels on account of the narrowness of the notch. If a suitably shaped test specimen could be found, which would give comparative results with the standard and would be at the same time easier to machine, it would perhaps be advantageous to adopt it.

A piece of hot-rolled machinery steel $\frac{1}{2}$ by 3 by 28 in. was selected from stock and cut into lengths of 3 in. The analysis of this material was as follows:

Carbon	0.134 per cent
Manganese	0.39 "
Silicon	0.015 "
Sulfur	0.021 "
Phosphorus	0.017 "

The pieces when cut to length were annealed by heating slowly to 875° C. They were held at this temperature ten minutes and allowed to cool slowly in the furnace over a period of five or six hours to room temperature. From each of the

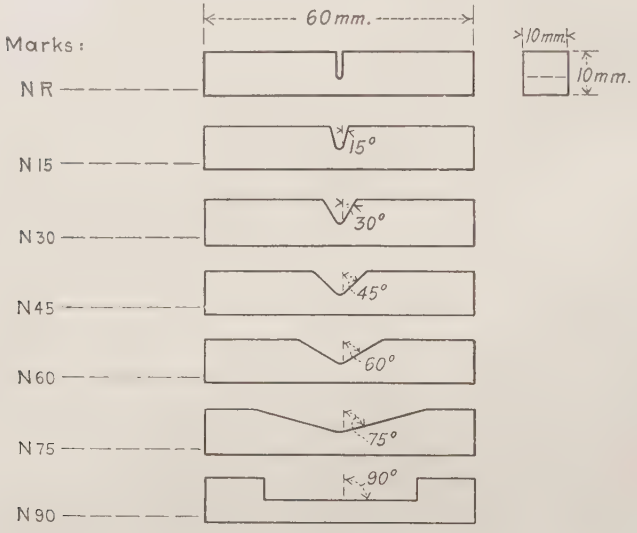


FIG. 14.—Charpy Test Specimens.

annealed pieces six transverse Charpy test specimens were machined as indicated in Fig. 14. The specimens were all taken out along the original direction of rolling of the bar. These specimens were tested in the Charpy machine in the usual manner and the results are shown in Table IV. The angle of bend shown represents the amount the specimens bent before rupture. These figures were obtained by putting the broken ends together after fracture, and measuring this angle.

An examination of Table IV will show that the widening out of the notch did not greatly affect the average figures for

resistance to shock until the slope of the side of the notch exceeded 45 deg. The figures for resistance to shock of the 15-deg. specimens are somewhat higher than those for either the regular or the 30-deg. specimens. This difference, however, is hardly greater than may be accounted for by experimental error. It will be noticed in this connection that the Brinell hardness numbers of these specimens are the lowest of any, and, in general, the lower the hardness is, the greater is the resistance to shock. Figures for the angle at the bend have been recorded, though these are somewhat uncertain, owing to the difficulty of satisfactorily placing together the two broken ends of the specimen.

TABLE IV.—EFFECT OF SHAPE OF NOTCH ON CHARPY IMPACT TESTS.

Specimen No.	Charpy Impact, ft-lb. per sq. in.			Angle of Bend, deg.	Brinell Hardness Number; average of three specimens.	Remarks.
	Maximum.	Minimum.	Average of Six Specimens.			
N R	304.0	266.5	284.75	15.8	79.1	Regular specimen.
N 15°	345.9	283 0	315.70[a]	15.2	77.5	
N 30°	322.5	284.0	298.10	15.0	80.9	
N 45°	382.0	304.0	333.67	29.5	80.0	
N 60°	571.0	516.0	539.17	54.2	79.8	Fractured but did not separate.
N 75°	890.0	818.0	853.83	130.0	78.4	Fractured halfway through. Did not separate.
N 90°	830.0	806.0	814.5	136.0	78.4	Bent, but no fracture.

[a] Average of five specimens.

These figures, however, agree fairly closely with those for the shock strength.

Fig. 15 summarizes the results obtained in this experiment so that they may be seen at a glance. Both curves have the same abscissas, namely, the angle of slope of the side of the notch, while the ordinates for curve *A*, are foot pounds per square inch, and for curve *B* are angle of bend in degrees. From these curves it may be stated that resistance to shock and angle of bend are not affected in Charpy impact tests by cutting away the side of the notch up to as much as 30 deg. By increasing the slope of the side of the notch from 30 to nearly 90 deg., or at least to 75 deg., the resistance to shock and the angle of bend are rapidly increased.

The results of the tests of the specimens with a 90-deg. slope—that is, the specimens with no notch whatever—do not fall on the curve, for the point of fracture was not located and the pieces did not fracture.

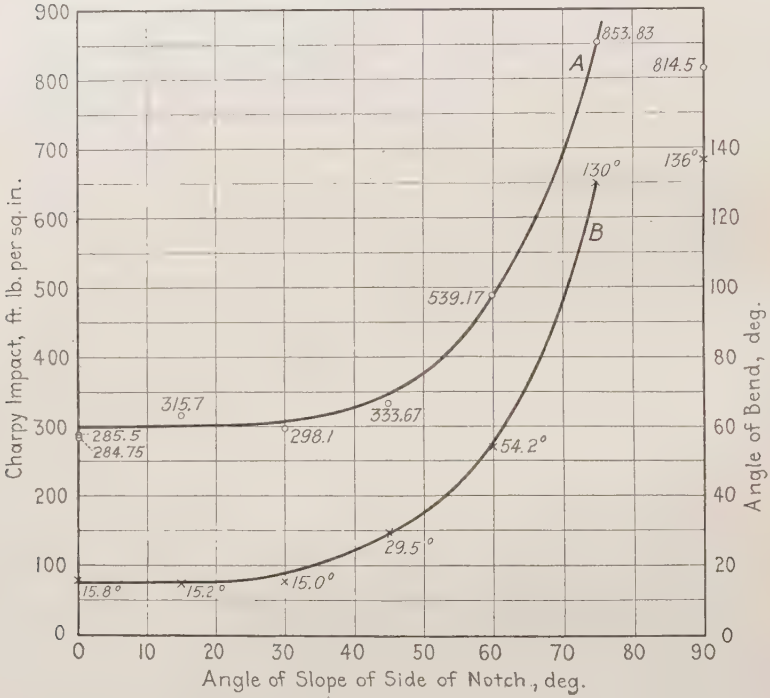


FIG. 15.—Effect of Shape of Notch on Charpy Impact Tests.

CONCLUSIONS.

Although the Charpy impact test is little used in this country it is believed that the above experiments indicate its value in determining the toughness or shock-resisting qualities of heat-treated steel.

The results given with tests of alloy steels prove conclusively that no reliable comparisons can be made by breaking single test specimens. From four to six similar specimens should be broken and the average value obtained before comparisons are

made. In all four experiments a standard test specimen 10 by 10 by 60 mm. was used, the span being 40 mm. Although the notch is usually made 1.3 mm. wide by 5 mm. deep, the results of tests on the effect of the shape of notch indicate that the sides may be flared to an angle of 30 deg. without introducing an appreciable error.

The maximum resistance to shock appears to be reached in quenched steels by drawing or tempering at 650° C. or just under the Ac1 point. Water-quenching after drawing appears to reduce the shock strength of heat-treated steels, the cause of which is unknown but appears to be due to internal stresses set up by unequal cooling. The forging of steel appears to give it a toughness which cannot be obtained by heat treatment alone.

A large variety of ordnance steels has been subjected to this Charpy impact test at the Watertown Arsenal during the past year, and some important improvements have been made as a result of the data obtained.

In conclusion, I wish to express my thanks to Mr. J. H. Nead, metallurgist at the Watertown Arsenal, for assistance rendered in the preparation of this paper.

MICROGRAPHIC DETERMINATION OF SURFACE DECARBURIZATION IN HEAT-TREATED STEELS.

By J. G. Ayers, Jr.

SUMMARY.

The object of this paper is to call attention to the fact that surface decarburization frequently exists in heat-treated steel parts to such an extent that in some cases the object of the heat treatment is more or less defeated, and to point out a micrographic method for investigating this defect in detail.

The procedure followed was to normalize the specimens to be examined in an atmosphere of nitrogen, to prevent any decarburization in this preliminary step. The specimens were then polished and etched in the customary way. A cross-section of the specimen was first prepared and a photomicrograph taken, from which a preliminary study of the decarburization was made. Further photomicrographs were then taken of longitudinal sections to study in detail the effect in each zone.

The conclusions warranted are that the subject should be more fully investigated than has been done in the past, as the evidence shows very plainly what a serious effect it has on surface hardness. The metallurgist should, therefore, investigate each type of steel employed and determine at what stage in the process of manufacture this condition is produced, so that he may intelligently take such steps as will remove it from the finished article. In cases where some of these conditions are beyond his control, the grinding limits may be so set that the decarburized portion is removed, and in this connection the metallurgist should play a more conspicuous part than he has in the past.

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By J. G. Ayers, Jr.

When heat-treated steel parts are ground two objects are accomplished: the part is reduced to the finished size, and a suitable finish is given to the surface. When parts are to be subjected to sliding or rolling contact and their ability to resist wear is a desideratum, a third object should be accomplished by the grinding process. This is the removal of any decarburized surface envelope or skin which has been occasioned by the heat treatment, or any prior process in the manufacture of the steel. Under ideal conditions for heat treatment, this decarburization could be reduced to a minimum; but in many commercial hardening operations it is more or less present and is quite frequently ignored. To ascertain to what extent it takes place was the object of the investigation described in this paper.

The microscope was chosen as the most satisfactory means of investigation, for the chemical analysis of successive layers involves a very laborious and painstaking preparation of the samples. It gives, to be sure, a closer approach to the absolute carbon content than can be obtained by the microscope, but this is not a serious drawback, as the relative amount of decarburization is all the information generally desired.

In order, however, to examine micrographically the carbon content of a steel, it is necessary to submit the steel to a thermal treatment that will impart a structure in which the carbon-bearing portion or pearlite can be readily resolved from the other constituents. This necessitates heating the specimen to well above its critical range, followed by slow cooling, to obliterate the structure produced by any previous treatment, and to give the most favorable conditions for the complete precipitation of the pearlite. Unfortunately, this process of heating to about 1000° C. is in itself conducive to surface decarburization, and we cannot be absolutely certain whether our conclusions are based upon the condition of the steel as regards carbon content before or after

the normalizing treatment. In other words, we are producing possibly the very condition for which we are searching. It is, of course, customary to pack the specimen in some suitable material to prevent this effect, and while this may answer the purpose satisfactorily enough where the extreme surface is not under investigation, it will not do for the purpose under discussion, for the reason that the slightest access of gas included or generated in the packing material may produce an undesired effect. In order to entirely eliminate this condition and to be certain that any decarburization noted had occurred in the heat-treatment or some previous treatment and not in the normalizing, all specimens were normalized in an atmosphere of nitrogen gas. This was readily accomplished by placing the specimen in a small gas-tight refractory tube, through which a stream of nitrogen passed from a tank of the compressed gas. This tube was inserted in a small Haraeus tube furnace to heat the specimen to the desired temperature.

The procedure adopted consisted in first preparing a photomicrograph of a cross-section of the specimen to make a preliminary survey under a medium low power. From a study of the photomicrograph of this cross-section, an idea was obtained of the depth of the decarburized zone. As the transition from the zone of minimum to that of maximum carbon content was very rapid in the cross-sectional view, additional longitudinal sections were prepared at such depths below the surface as the cross-section photomicrograph suggested would be of value. From these longitudinal views which traversed zones of fairly constant carbon content, an estimate of the degree of decarburization could be obtained.

Fig. 1 shows the location of the sections of which Figs. 2 to 6, inclusive, are photomicrographs.

Fig. 2 is a photomicrograph showing a cross-section of the steel including the extreme edge of the specimen. Its location is *abcd*, Fig. 1. The scale across the center of Fig. 2 shows that decarburization extends to a depth of about 0.015 in. About 0.020 in. below the surface the normal carbon content is undisturbed and hence from this an opinion of the degree of decarburization can be formed, although this decarburization is much better shown in the longitudinal views to follow.

Fig. 3 is a photomicrograph of a longitudinal section 0.003 in. below the surface. It was taken on the plane located by *efg*, Fig. 1, and shows that the degree of decarburization at this depth was very marked. This evidence is in accord with the cross-sectional view just observed above. Sorbitic pearlite[1] occupies only about 25 per cent of the total surface.

Fig. 4 shows a longitudinal section 0.007 in. below the surface on the plane *hmn*, Fig. 1. It does not show such marked decarburization, sorbitic pearlite occupying about 45 per cent of the total surface.

Fig. 5 shows a longitudinal section 0.013 in. below the surface on the plane located by *opr*, Fig. 1. It exhibits still

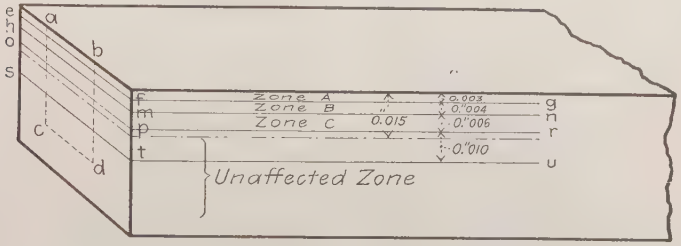


FIG. 1.—Location of Sections of which Photomicrographs Were Taken.

less the effects of decarburization, sorbitic pearlite occupying about 70 per cent of the total surface.

Fig. 6 shows a longitudinal section 0.023 in. below the surface. It is on the plane located by *stu*, Fig. 1, is well within the unaffected zone and is representative of the normal carbon content of the piece. Sorbitic pearlite occupies about 80 per cent of the total surface.

We are now in a position to form an estimate of both the extent and the degree of the decarburization. In estimating the amount of carbon micrographically, care should be exercised to determine exactly upon what micro-constituent the determination is based. If the experimenter's normalizing treatment does not result in pearlite of eutectoid carbon content a factor should

[1] The term "sorbitic pearlite" in this paper is applied to any of the phases of pearlite or to sorbite itself.

be determined for each type of steel handled, and for the particular normalizing treatment; otherwise, determinations will often be wide of the mark. This is due to the fact that different rates of cooling in the normalizing treatment result in different phases of pearlite, including sorbite itself. After a factor has once been obtained by chemical check no repetitions need be made.

The specimen under discussion showed about 80 per cent of sorbitic-pearlitic areas in Fig. 6. If we consider them as pearlite containing 0.85 per cent carbon, this view would depict a spot of 0.80 × 0.85, or 0.68 per cent carbon. Chemical analysis gave

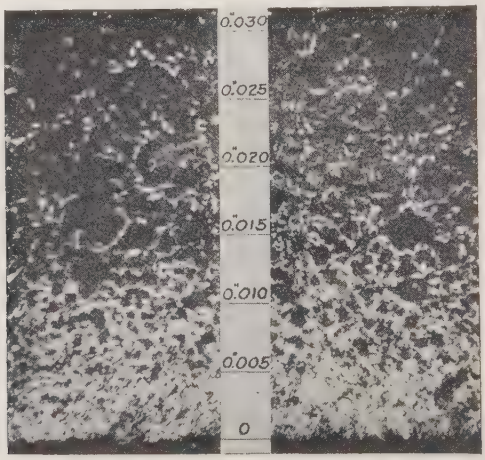


FIG. 2.—Photomicrograph of Section *abcd* (Fig. 1).

0.45 per cent. This illustrates the point dwelt upon above. If on the other hand we assume that 80 per cent of sorbitic pearlite corresponds in this particular case to 0.45 per cent carbon, we have a more consistent factor to work by, and hence we deduce the following:

Fig. 3, representing the maximum carbon content of zone *A*, Fig. 1, shows 25 per cent sorbitic pearlite, or 0.14 per cent carbon;

Fig. 4, representing the maximum carbon content of zone *B*, shows 45 per cent sorbitic pearlite, equivalent to 0.25 per cent carbon;

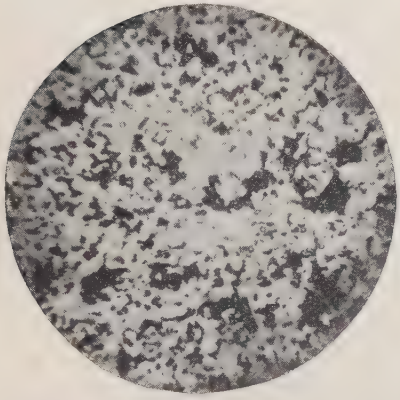


FIG. 3.—0.003 in. below Surface; Sorbitic Pearlite, 25 per cent.

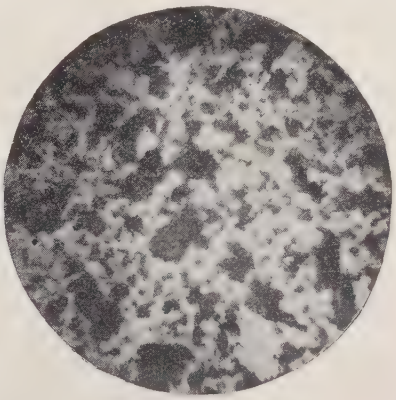


FIG. 4.—0.007 in. below Surface; Sorbitic Pearlite, 45 per cent.

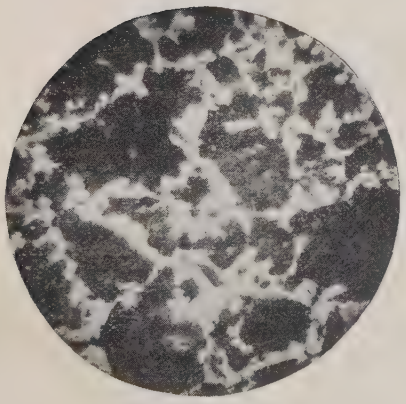


FIG. 5.—0.013 in. below Surface; Sorbitic Pearlite, 70 per cent.

FIG. 6.—0.023 in. below Surface; Sorbitic Pearlite, 80 per cent.

PHOTOMICROGRAPHS OF LONGITUDINAL SECTIONS AT VARIOUS DEPTHS BELOW SURFACE. MAGNIFICATION, × 200.

Fig. 5, representing the maximum carbon content of zone *C*, shows 70 per cent sorbitic pearlite, equivalent to 0.39 per cent carbon.

If on the other hand we desire to ascertain only the relative degree of decarburization in each zone, as compared with the original content, we need no recourse whatever to chemical analysis. Inasmuch as Fig. 6, which represents the zone unaffected by decarburization, contains 80 per cent sorbitic pearlite, zones *A*, *B* and *C*, containing 25, 45 and 70 per cent sorbitic pearlite respectively, have lost by decarburization about 69, 44 and 12.5 per cent, respectively, of their original carbon content.

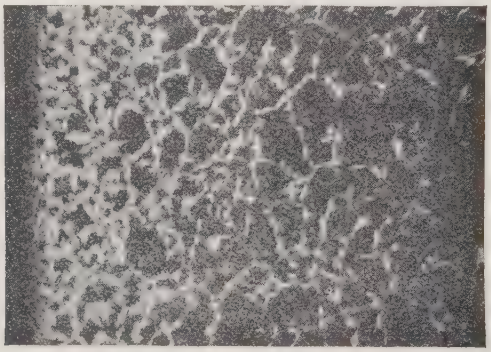


FIG. 7.—Small Hot-Rolled Section as Furnished by Mill.
Magnification, × 75.

From an estimate of this nature, the metallurgist can ascertain just how much should be ground from the piece to satisfy his particular requirements. In the case of the piece just examined, it is manifestly evident that at least 0.015 in. must be removed from the surface to entirely remove all traces of decarburization. It is obvious that this piece has not been properly manipulated either in the heat treatment or the mill. This conclusion gives rise to the question as to whether the cause lies with the mill or the heat treatment, or possibly a combination of both. This can of course be readily determined by a micrographic examination before any heat treatment has been attempted. Results will no doubt differ considerably for different types and sections of steel finished by different processes and made for diverse purposes.

Fig. 7 shows a cross-section of a small hot-rolled section as furnished by the mill. With the exception of normalizing, it has been given no further treatment. It shows evidences of decarburization to a depth of about 0.010 in. Fig. 8 shows a piece of

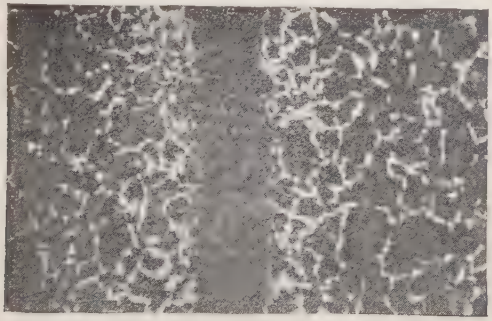


FIG. 8.—Same Steel as Fig. 7 Heated to 790° C. for 3 Hours and Quenched in Oil. (Two Portions of the Specimen are shown with Decarburized Edges Facing each other.) Magnification, × 70.

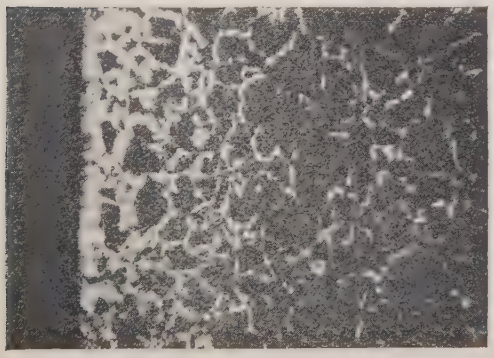


FIG. 9.—Same Steel as Fig. 7 Heated to 790° C. for 9 Hours and Quenched in Oil. Magnification, × 75.

the same steel. All the zone decarburized by the mill was removed and the piece was then heat-treated according to the practice in vogue for its particular application. This consisted of heating to about 790° C. for 20 minutes and then quenching

in oil. In this case, however, the time in the furnace was purposely prolonged to a period of 3 hours to accentuate any decarburizing effects. It will be noted that the decarburized zone is not over 0.005 in. and hence less than that produced by the mill. Another piece of this same stock similarly prepared but kept in the furnace 9 hours is shown in Fig. 9. Here it will be observed that the decarburization is about as deep as that produced by the mill. We therefore note that in this particular instance the mill was the most potent factor in causing decarburization, as that caused by heat treatment was not very marked, except where the duration of the time in the furnace was abnormally prolonged.

In conclusion, it may be stated that surface decarburization frequently exists in heat-treated steel parts, due either to the mill practice or to the heat treatment itself. From whatever cause, it should be investigated by the metallurgist and its extent ascertained. The microscope is undoubtedly the most efficient means of accomplishing this purpose in hypo-eutectoid steels. When the exact cause and extent have been determined, means may be provided for correcting it—at least to a certain extent. In the grinding operation, however, there sometimes lies the opportunity of entirely removing its deleterious effects by a judicious determination of the grinding limits, and in this the metallurgist should play a more conspicuous part than he has in the past.

MOLTEN ZINC AS A REAGENT FOR THE MACROSCOPIC ETCHING OF IRON AND STEEL

By Jesse L. Jones.

SUMMARY.

The samples are immersed in molten zinc for varying periods, low-carbon steels requiring a longer time than high-carbon steels. The adherent dross and metallic zinc are removed partly by mechanical means and partly by the aid of dilute sulfuric acid. Defects in the steel are thus very plainly disclosed, porous areas being attacked to a marked degree while the non-porous areas are scarcely attacked at all.

High-carbon steels are more readily attacked than low-carbon steels, and lack of uniformity may be thus ascertained as well as the depth and character of case-hardening.

High-speed steels are etched in a remarkable manner that indicates that the constituent metals are seldom if ever perfectly alloyed. The deeply etched spots are probably very high carbon areas that are vigorously attacked by the molten zinc.

MOLTEN ZINC AS A REAGENT FOR THE MACROSCOPIC ETCHING OF IRON AND STEEL.

By Jesse L. Jones.

The microscopic examination of steels after polishing the surface of small samples and then etching with various reagents, has been brought to a high degree of excellence. Not so much work has been done, however, in rendering visible on a large scale the porosity, lack of homogeneity in composition, hair cracks, blisters, seams, overlaps, slivers and other defects in steel that the rolling or forging treatment has rendered difficult to detect by ordinary visual inspection.

A large user of machinery steel forgings recently obtained, from various steel works, a number of 15-in. ingots and sawed them in half longitudinally. The sawed surfaces when polished showed hundreds of large air bubbles and innumerable small ones, as well as hair cracks in the case of ingots that had stuck in the molds and had not been allowed to shrink freely for this reason. The subsequent mechanical treatment that such ingots receive never altogether removes the porosity and other defects due to the enclosed or occluded air and gases in them. This was proved experimentally by spinning a glass rod about a platinum wire 0.040 in. in diameter, so as to give it an impervious coating of glass. The wire thus coated was heated electrically to a red heat in a vacuum and bubbles of air or gas continued to force their way through the glass for several days.

In the making of low, medium and high-carbon steels in the open-hearth furnace there is quite a divergence in practice, some makers adding to the steel while in the furnace the ferro-alloys used, while others add them to the steel as it runs into the ladle. Some makers recarbonize while others "bring down" the pig-iron and steel-scrap mixture to the required carbon.

In the case of alloy steels, such as high-speed tool steels which contain notable amounts of tungsten and other metals with high melting points, the tendency to lack of uniformity is more marked than in ordinary steel. These steels are usually

made by the crucible process. Plumbago crucibles holding from 90 to 160 lb. (these may be taken as the minimum and maximum sizes used) are placed in regenerative furnaces accommodating ten crucibles. These are previously charged with the steel mixture, which consists of sheared muck bar (made from low-phosphorus or Bessemer pig iron), ferro-tungsten, ferro-chromium, ferro-manganese, ferro-vanadium, cobalt, etc. The crucibles are tightly sealed so as to keep out the air as much as possible. An effort is made to keep the temperature at the bottom of each crucible at about 3100° F., or several hundred degrees higher than the top, in order to "lift" the tungsten from the bottom of the crucibles, its high specific gravity and high melting point rendering this necessary. To prevent an undue amount of carbon being taken up by the steel, the crucibles are clay-lined or clay-washed and a quantity of crushed soft red brick or similar material is added to glaze over the surface of the crucibles. Even with these precautions the carbon in the various crucibles is different. It has been found that if the crucibles are poured one by one into an ingot mold, a "calico" ingot or one of varying carbon content is produced. Hence the crucibles are emptied into a steel ladle and this is bottom-tapped into the ingot mold, thus giving a more homogeneous ingot.

It is evident from the above review of the processes of steel making that they are far from perfect, and while chemical analysis, microscopic examination and the various physical tests such as tension, Brinell hardness, etc., are more or less satisfactory for the inspection of steel, any improvement in the methods of macroscopic examination would be very desirable. Such an improvement seems to have been found in etching the steel with molten zinc.

The writer had occasion recently to ascertain the relative value of ingot iron, Norway iron and open-hearth steel for making pots for hot galvanizing. Bars 10 by $1\frac{1}{4}$ by $\frac{5}{16}$ in. of each of these materials were suspended in the molten zinc in a 14-ft. galvanizing pot, by means of iron wires. The samples were placed at the end of the pot because there was no firebox at that point, and hence if one sample happened to be nearer the end of the pot than another it would not for that reason be subjected to a higher temperature. Later, similar samples of carbon tool

steel and high-speed steel were added. On taking the samples from the molten zinc the mass of dross surrounding them could be broken off while still hot by blows from a hammer, and the coating of metallic zinc removed by immersion in dilute sulfuric acid, which does not attack the steel as long as any zinc remains. Table I gives the results of this experiment.

The appearance of the samples after treatment is shown in Fig. 1, *N* being the ingot iron, *O* the Norway iron, *H-3* the high-speed steel, and *R* the carbon tool steel.

The high-speed steel had its surface deeply pitted and grooved at some points, but the rest of the surface was scarcely attacked at all. The spots that were attacked are probably areas high in

TABLE I.—LOSS OF BARS ON IMMERSION IN MOLTEN ZINC.

Material.	Time of Immersion, hours.	Total Loss, per cent.	Loss per Square Inch in 24 Hours, per cent.
Ingot Iron	168	2.95	0.012
	336	6.78	0.014
	672	12.79	0.013
Norway Iron	168	3.73	0.016
	336	7.46	0.016
	672	14.22	0.014
Open-hearth Steel	168	3.93	0.017
	336	7.57	0.016
	576	12.80	0.016
	720	15.76	0.016
High-Speed Steel	24	7.39	0.199
Carbon Tool Steel	48	21.57	0.348

carbon, due to imperfect alloying of the ferro-tungsten and other additions. These additions are usually high in carbon and also of high melting point. Hence they do not diffuse very rapidly and the resulting mixture may not be homogeneous.

The carbon steel was not only eaten away to a high degree, but it also showed a curious shelly structure, the edges being cut away and apparently concentric layers of steel exposed to view as if the steel during the rolling process had not been sufficiently plastic to fill out the grooves in the rolls, and these edges, either because of minute fissures or on account of not being sufficiently compressed to remove porosity, had readily dissolved in the molten zinc.

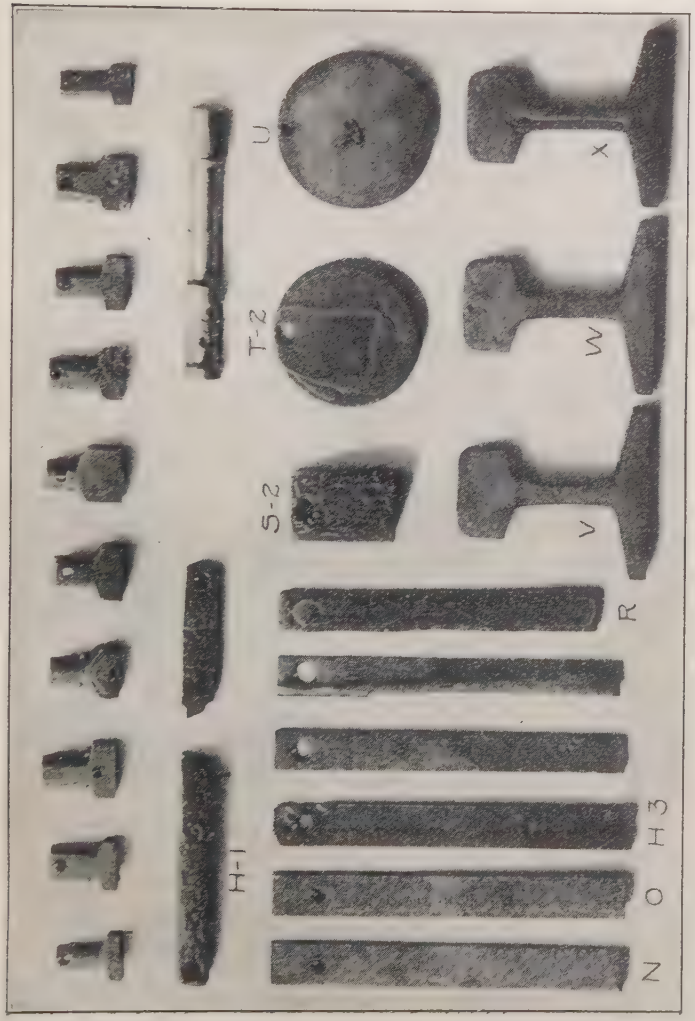


FIG. 1.

Fig. 1 also shows a number of other more or less interesting samples. The sections of Bessemer steel rails (*V*, *W* and *X*) seemed perfectly solid before etching, but after etching the web part of each sample could be seen to be spongy, the section *X* being especially so.

A defective axle that failed in service (*U*) showed a pipe after etching.

A piece of die steel (*S-2*) was rejected because one surface was badly blistered. On placing this sample in the molten zinc

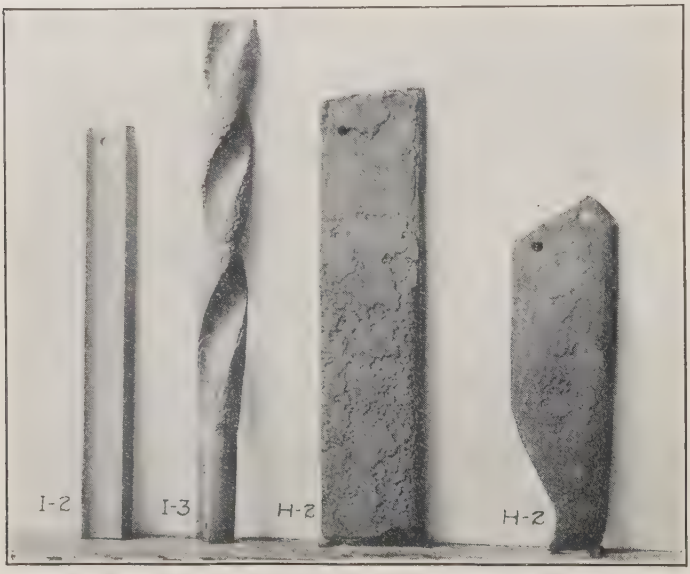


FIG. 2.

the blistered portion was deeply etched, as seen in the photograph, while the other surfaces remained smooth.

The bar of special steel from which sample *T-2* was taken had been very unsatisfactory, the milling cutters made from it nearly all failing. It seems to be non-uniform in composition.

The high-speed tool steel (*H-1*) is interesting, as the highly hardened end has not been attacked by the molten zinc, while the other end is eroded in the usual manner. The intense heat to which the point of the tool was subjected in hardening, no

doubt, removed the carbon superficially, so that the steel was able to act like a low-carbon steel and thus resist the molten zinc.

Fig. 2 shows an etched sample of high-speed steel drill stock (*I-2*) that was unsatisfactory and a defective drill (*I-3*) made from this stock.

Two portions of a large high-speed steel planer tool that broke in service are marked *H-2*. Assuming the eroded portions to be brittle high-carbon areas, unable to resist shock under stress, the failure of the tool is understood.

While no specific tests have been made, the readiness with which the molten zinc dissolves highly carbonized steel indicates that this method of testing can be used to show the depth and character of the surface hardening of low-carbon steel that has been case-hardened.

A few rather unsatisfactory trials have been made of etching nickel steel in molten tin.

The use of metallic mercury as an etching fluid and the extension of this method of testing to non-ferrous metals is under consideration.

This very incomplete work is described in hope that other investigators may be able to carry it further and develop it into a useful method of macroscopic investigation.

DISCUSSION.

Mr. Campbell.

MR. WILLIAM CAMPBELL (*by letter*).—In discussing Mr. Jones' paper, in which he states that while microscopic examination has been brought to a high degree of excellence, not much work has been done macroscopically, I should like to point out that the subject has in reality had a great deal of attention, as shown by the papers by Ast and Frêmont.

Having had occasion recently to look up the question of the macroscopic etching of iron and steel, I gained quite a little information which I venture to present in the form of a bibliography with an occasional abstract, as the most convenient method.

Frémont points out that the artistic application is very ancient, for the art of damascening was well known to the Egyptians 3600 years ago. A few dates in regard to the industrial application are of interest:

1779.—Jean-Jacques Perret pointed out that Damascus steel could be tested by etching.

1783.—Bergman, etching with sulfuric acid, distinguished iron from steel.

1850.—Edwin Clark ("The Brittania and Conway Tubular Bridges," London, 1850, p. 632) showed the difference between hand and machine rivetting by etching sections of the two.

1872.—Van Ruth (*Berg- und Hüttenmännische Zeitung*, Leipzig) tested the quality of iron by etching.

1873.—Kick showed the structure of welded material.

1875.—Bruno Kerl recommended etching in his Outlines of Metallurgy.

1878.—Von Kerpely in his work on rails showed numerous etched sections of iron and steel.

1886–1894.—Papers by Ledebur, Stein, Wedding, Martens and others appeared in *Stahl und Eisen*.

1893.—Martens, in "The Microstructure of Ingot Iron in Cast Ingots," Chicago meeting, American Institute of Mining Engineers, August, 1893, at the International Engineering Congress, gives examples of macrostructure.

1896.—Tetmajer, in his work on Basic Rail Steel, notes that etching tests are very important.

1896.—Committee appointed by the Board of Trade to inquire into the loss of strength of steel rails through use on railways. Roberts-Austen's appendix to the report (H. M. Stationery Office, 1900) discusses both the macro- and microstructure.

Mr. Campbell.

The following are the most important recent references:

1906.—Ast, on the international researches in macroscopic examination (etching tests) of iron, International Association for Testing Materials, Brussels Congress, 1906. Report on Problem 36.

First shows the historical development and then discusses the present position of this subject. Numerous photographs illustrate the report. The etching materials are preferably:

1. Dilute HCl, HNO_3 and H_2SO_4 up to 30 per cent acid.
2. 20 g. of I and 30 g. KI with 1000 g. of Water (Tetmajer).
3. HCl in alcohol up to 30 per cent (Martens and Heyn).
4. 10-per-cent copper-ammonium chloride (Martens and Heyn).
5. 10-per-cent ferric chloride.

First wash the surface with alcohol or alcoholic solutions of KOH or NaOH. After etching, wash well and dry with alcohol, then ether.

Etching with acids enables impurities to be detected. Iodine or copper-ammonium chloride enables the more highly carburized portions to be detected as well.

1906.—Heyn (*a*) International Association for Testing Materials. Brussels Congress, Paper No. 6.

(*b*) "Bericht über Ätzverfahren zur Macroscopischen Gefügeuntersuchung des Schmied-baren Eisens," *Mitt. aus dem Materialprüfungsamt*, 1906, H. 5.

1908.—Ch. Frémont. "Essais des fers et des aciers par corrosion," *Rev. de Metallurgie*, Vol. V, p. 649. Discusses artistic application with illustrations. Refers to Ast and Heyn. Gives numerous illustrations and shows method of taking the photograph. The following solutions are given.

1. H_2SO_4 ..... 20 cc. per 100 cc. of water.
2. HNO_3 ..... 15 cc. } per 100 cc. of water.
   HCl ..... 8 cc. }
3. HNO_3 ..... 67 cc; HCl 33 cc.
4. Pure HCl.
5. Copper-ammonium chloride .... 10 g. per 100 cc. of water.
6. Chloride of iron ..... 10 g. per 100 cc. of water.
7. Bichloride of mercury ..... 5 g. per 100 cc. of water.
8. I ..... 10 g. } per 100 cc. of water.
   KI ..... 20 g. }

1908.—Wawrziniok. *Handbuch des Materialprüfungswesens.* (Julius Springer.) Discusses macroscopic examination, p. 545. Gives examples of rails, bending and compression test pieces, forgings, etc.

1908.—Report of the Tests of Metals, Watertown Arsenal, shows sections of steel rails. In the 1909 report, which deals largely with the investigation of steel-rail ingots and derivative shapes, a mass of information as to macrostructure is found. The etching agent was iodine, which reveals the structure very strongly.

Mr. Campbell. 1912.—Bauer and Deiss. "Probenahme und Analyse von Eisen und Stahl" (Springer).

Advocates Heyn's solution, copper ammonium chloride, 10 g. in 120 cc. of water, for 1 to 2 minutes for macroscopic etching. Examples of cast and rolled material (pp. 77, 79).

1913.—Preuss. "Die praktische Nutzanwendung der Prüfung des Eisens durch Ätzverfahren und mit Hilfe des Mikroskopes." (Springer.)

For macroscopic etching recommends Heyn's Solution. Gives examples of wrought iron, steel, welding, punching, riveting, etc.

1914.—Rosenhain. "An Introduction to the study of Physical Metallurgy." (Van Nostrand.) Discussing the development of structure (p. 34), speaking of heat tinting, first developed by Stead (*Journal Iron and Steel Institute*, 1900, Vol. II, p. 137), says this method has been employed for a variety of purposes but primarily for detecting the distribution of phosphorus in iron and steel. He mentions the use of solutions of the copper salts and says that Giolitti's method for the copper-tin alloys (*Gazetta Ital. Chem.*, 1906, 36 II, p. 142; 1908, 38 II, p. 352), the very gradual deposition of metallic copper by a process of electro-chemical replacement, has been successfully used for iron and steel.

1914.—Rosenhain and Haughton. "A New Reagent for Etching Mild Steel." *Journal Iron and Steel Institute*, 1914, Vol. I, p. 515. This reagent, which brings out very clearly the segregation of the phosphorus, consists of:

Ferric chloride	30 g.
Hydrochloric acid	100 cc.
Cupric chloride	10 g.
Stannous chloride	0.5 g.
Water	1 liter

1915.—Stead. "Iron, Carbon, and Phosphorus," annual meeting of the Iron and Steel Institute, 1915, gives a new method of detecting variations of phosphorus in iron and steel. The composition of the reagent is as follows:

Cupric chloride	10 g.
Magnesium chloride	40 g.
Hydrochloric acid	20 cc.
Alcohol	to 1000 cc.

The salts are dissolved in hot water and the solution made up to 1000 cc. with alcohol. The specimen is covered with a thin layer of the reagent and must not be immersed in a bath of the liquid. The etching is repeated until the structure is developed.

While the above reagents were developed primarily for microscopic work, they are also useful for macroscopic examina-

tion. It is not out of place to mention here another method of investigating macrostructure which has yielded excellent results. Mr. Campbell.

Sulfur Printing.—A piece of smooth silver bromide paper is moistened with 3-per-cent sulfuric acid and carefully applied to the polished surface of the section. In about 30 seconds the paper is withdrawn, washed and fixed in hyposulfite. The sulfur-rich areas are colored dark and a print showing segregation is thus obtained.

R. Baumann. *Metallurgie*, 1906, p. 416.
Stead. "Sulphur and Iron," Staffordshire Iron and Steel Institute, March, 1908.
Hatfield. "Cast Iron," p. 71, shows sulfur prints of pig-iron.

Finally, as an example of what can be done both by iodine etching and by the method of sulfur printing, reference should be made to the work of Comstock in the Rail Reports issued by the Titanium Alloy Manufacturing Co., Niagara Falls, 1913–1914.

Mr. H. S. Rawdon (*by letter*).—The microscopic study of alloys, in general, and of steel, in particular, has made clear and emphasized the fact that metals are not to be considered as homogeneous substances similar to glass or other amorphous bodies, but rather that they are to be placed in the same category as rocks and to be examined from the same viewpoint. The one thing most apparent to the microscopist of metals, the heterogeneity and non-uniformity of alloys, is far from being realized and comprehended by users in general. Mr. Rawdon.

The microscopic method of study has its limitations which the metallographist realizes most strongly of all concerned, and, as has been pointed out by Mr. Jones, it is in the examination of metals with the unaided eye, or at relatively low magnifications, that improvements and new methods of examination are most urgently needed.

Of course there are well-known methods for the determination of segregation and non-homogeneity of iron and steel in general, for example, "sulfur prints" and etching tests for carbon and phosphorus segregation, etc. These, however, require the preparation of fairly well-polished plane surfaces which, in the case of large specimens, is laborious and expensive. The

Mr. Rawdon. method here proposed suggests that the same features may be shown on the sample in its usual form.

At the Bureau of Standards, one line of work being carried on at present has for its object the determination of how closely the various metalloids follow each other in segregating, upon cooling from a molten state. To put the question in a concrete form: How closely can we rely upon the "sulfur print" as a record of carbon and phosphorus segregation? Of course, theoretically they act the same. It will be very helpful to know whether Mr. Jones has attempted to correlate the results of his method of zinc-etching with some of the older and better-known ways of macroscopic examination, and if so, how they compare. Are there any features not shown by the other methods that are revealed by the zinc etching?

In sawing up rails and other similar rolled stock, concentric layers similar to those noticed by Mr. Jones in the case of the zinc-etched high-carbon steel have frequently been noticed upon the sawed surface. In a few cases these have been seen in sections of small steel ingots which have received no work. So far as is known to the writer, these concentric layers have never been found to be associated with a microstructure which is markedly different from that of the remainder of the sample.

As it seems to the writer, the one advantage that should be emphasized for this method of examination is the fact that samples may be examined *as a whole* and no laborious preparation of large plane sections is necessary.

FATIGUE OF COPPER ALLOYS.

By Ernst Jonson.

SUMMARY.

Experience has shown that copper alloys are subject to cracking under certain conditions of stress and exposure. In order to determine the magnitude of the stress at which cracking is liable to occur, a series of long-time tension tests has been started, in which the test specimens are exposed to the action of ammonia while under stress. In order to discover which alloys are subject to cracking, all the common alloys will be included in this series of tests.

Results have been obtained from the following alloys: Manganese bronze, naval brass, Muntz metal and gun metal. All the specimens of these metals cracked when the stress reached a value between 20,000 and 27,000 lb. per sq. in. Specimens of manganese bronze exposed to high stresses for a long time, but protected from corrosion, did not crack. Other specimens of manganese bronze, which were corroded without being under stress, were not injured thereby.

The following conclusions have been drawn from the tests:

Excessive stress alone does not injure copper alloys, nor does corrosion alone, or corrosion accompanied by moderate stress. Corrosion, accompanied by prolonged stress exceeding 20,000 lb. per sq. in., is liable to cause cracking in any of the above-mentioned alloys. We must therefore regard 20,000 lb. per sq. in. as the practical ultimate strength of copper alloys, and the working stress must be taken as a safe fraction of this ultimate strength.

FATIGUE OF COPPER ALLOYS.

By Ernst Jonson.

In previous papers[1] the writer has pointed out that the occurrence of so-called season cracking in brass rods is conditioned by (1) high initial tensile stress in the surface of the rod, and (2) by corrosion of the metal; also, that a brass rod may develop cracks of the same nature as season cracks when stressed beyond the initial yield point by an external load, instead of having initial stress set up by the cold drawing of the rod, and that such cracking is not peculiar to one kind of brass but that all kinds of forgible brass are subject to it.

At the time of the previous writing, the following brasses were tested and cracked when etched with ammonia, while under stress greater than the initial yield point: Cast manganese bronze from two foundries, plain-extruded manganese bronze, rolled-and-drawn manganese bronze, plain-extruded naval brass, drawn naval brass, and plain-extruded Muntz metal. No cracking has been observed where high stress and corrosion did not co-exist. From these facts the conclusion was drawn that the ordinary ultimate strength of brass is developed only when the metal is broken so quickly that cracks do not have time to develop, and that when a stress exceeding the yield point is applied for a considerable time the metal will fail by cracking.

The working hypothesis formed for the purpose of co-ordinating these facts and to guide further investigations is as follows:

In copper alloys there are microscopic flaws. When the tensile stress approaches the yield point these flaws are opened sufficiently to admit corrosion. This corrosion weakens the edges of the flaws and causes them to increase under the continued action of the stress, and thus a continuous crack is formed which gradually increases in depth. When the crack has so

[1] "Cracking of Drawn Brass," *Engineering Record*, April 11, 1914; "Troubles Experienced with Forgible Brass during the Construction of the Catskill Aqueduct," *Proceedings*, Am. Inst. Metals (1914).

reduced the cross-section that the stress on the remaining solid portion exceeds the ultimate strength, the rod breaks.

In order to more definitely determine the magnitude of the stress at which cracking begins another series of tests has been started. Each test was made as follows: A tension test specimen of 1 in. diameter and 8 in. length, with a reduced diameter of 0.8i n. and 6-in. gage length, was placed in the testing machine. A sheet steel cup with a 1-in. hole in its bottom was put on top

Test No. 2.—Manganese Bronze.

Test No. 4.—Naval Brass.

FIG. 1.—Specimens after being Tested.

of the lower grip so that the end of the test specimen passed through it, and the cup was filled with strong ammonia. Some absorbent cotton was tied to the test specimen so as to keep it wet by capillarity, and the cup and test specimen were covered by a conical paper hood so as to confine the ammonia fumes. A stress of 10,000 to 17,000 lb. per sq. in., increased by increments of about 3000 lb. per sq. in. at intervals of a day or more, was applied to the test specimen. Under the higher stresses the test

TABLE I.—RESULTS OF TESTS.

No. 1.—MANGANESE BRONZE, PLAIN EXTRUDED.

Time of Test.	Load, lb.	Stress, lb. per sq. in.
Nov. 23, 1914, 10.40 a. m., to 25, 10.40 a. m	5 000	10 000
Nov. 25, 10.40 a. m., to 27, 10.40 a. m	6 500	13 000
Nov. 28, 11.30 a. m., to 30, 10.15 a. m	10 000	20 000
Nov. 28, 12 noon, beam had dropped 0.5 in. and was raised.		
Nov. 30, 5 p. m., to Dec. 3, 10 a. m	11 500	23 000
Beam had to be raised every half hour until Dec. 5, 11.30 a. m.		
Dec. 5, 10 a. m., to 7, 10 a. m	11 500	23 000
Dec. 7, 1 p. m. to 1.30 p. m	13 500	27 000
Beam dropped and was raised continually until specimen broke by cracking. There were numerous cracks in immersed portion of test specimen. The last increment of load was evidently too great. A smaller increment probably would have started cracking.		

No. 2.—MANGANESE BRONZE, PLAIN EXTRUDED, HEATED TO NEAR MELTING POINT, QUENCHED IN WATER.

Dec. 18, 9 a. m., to 19, 9 a. m	7 000	14 000
Dec. 19, 9 a. m., to 20, 9 a. m	8 500	17 000
Dec. 20, 9 a. m., to 21, 9 a. m	10 000	20 000
Dec. 21, 9 a. m., to 24, 5 p. m	11 500	23 000
Beam raised continually.		
Dec. 24, 5 p. m. The specimen failed by cracking; two large cracks on opposite sides of specimen, each crack extending over half circumference; two minor cracks; all in immersed portion. See Fig. 1(*a*).		

No. 3.—MANGANESE BRONZE, PLAIN EXTRUDED.

Dec. 24, 9 a. m., to 28, 9 a. m	8 500	17 000
Dec. 28, 9 a. m., to 29, 9 a. m	10 000	20 000
Dec. 29, 9 a. m., to 30, 12 m	11 500	23 000
Dec. 30. Beam dropped and was raised continually. Specimen failed by cracking; one large and several small cracks in immersed portion.		

TABLE I.—RESULTS OF TESTS (*Continued*).

No. 4.—NAVAL BRASS, PLAIN EXTRUDED.

Time of Test.	Load, lb.	Stress, lb. per sq. in.
Mar. 25, 1915, 3.30 p. m., to 26, 3.30 p. m	5 000	10 000
Mar. 26, 3.30 p. m., to 29, 12 m	6 500	13 000
Beam raised Mar. 27, 9 a. m., and 28, 9 a. m.		
Mar. 29, 12 m., to Apr. 1, 1 p. m	8 500	17 000
Beam raised Mar. 29, 1 p. m., 3.30 p. m.; Mar. 30, 9 a. m., 1 p. m.; Mar. 31, 9 a. m., 1 p. m. Specimen removed from machine Apr. 1, 1 p. m. to 3 p. m.		
Apr. 1, 3 p. m., to 2, 12 m	10 000	20 000
Beam raised Apr. 1, 5 p. m.; 2, 9 a. m. and 10 a. m.		
Specimen failed by cracking, at 12 m., Apr. 2. Three large cracks, and some minor ones in immersed portion. See Fig. 1(*b*).		

No. 5.—MUNTZ METAL, PLAIN EXTRUDED.

Mar. 8, 1 p. m., to 9, 3 p. m	5 000	10 000
Mar. 9, 3 p. m., to 10, 3 p. m	6 500	13 000
Mar. 10, 3 p. m., to 15, 5 p. m	8 500	17 000
Beam raised Mar. 10, 5 p. m.; 11, 9 a. m., 11 a. m., 1 p. m., 5 p. m.; 12, 5 p. m.; 13, 5 p. m.		
Mar. 15, 5 p. m., to 24, 5 p. m	10 000	20 000
Beam had to be raised continually from Mar. 16, 9 a. m., to 22, 3 p. m.		
Mar. 24, 5 p. m., to 25, 3 p. m	11 500	23 000
Beam raised continually until 3 p. m. on Mar. 25, when specimen failed by cracking. One crack appeared near the upper thread and one near the lower thread. Several other cracks appeared on the immersed portion.		

No. 6.—GUN METAL (88 Cu, 10 Sn, 2 Zn) CAST.

Apr. 2, 1 p. m., to 5, 9 a. m	5 000	10 000
Beam raised Apr. 3, 9 a. m.		
Apr. 5, 9 a. m., to 8, 9 a. m	6 500	13 000
Beam raised Apr. 5, 12 m., 5 p. m.; 6, 9 a. m.		
Apr. 8, 9 a. m., to 14, 12 m	8 500	17 000
Beam raised Apr. 8, 10 a. m., 11 a. m., 12 m., 2 p. m., 3 p. m., 5 p. m.; 9, 9 a. m.; 10, 9 a. m.; 12, 9 a. m.; 13, 9 a. m.		
Apr. 14, 12 m., to 20, 9 a. m	10 000	20 000
Beam raised continually until Apr. 15; then Apr. 15, 9 a. m., 11 a. m., 3 p. m., 5 p. m.; 16, 9 a. m., 3 p. m., 5 p. m.; 17, 9 a. m.; 18, 9.30 a. m.; 19, 9 a. m.		
Apr. 20, 9 a. m., to 22, 12 m	11 500	23 000
Beam raised continually until failure occurred on Apr. 22, 12 m., by cracking. Crack near top.		

specimen continued to stretch for a considerable length of time, and in such cases this time was added to the interval between the additions of load.

The test specimen was examined before each increment of load was applied. When the first crack was seen the test was concluded, because experience has shown that a crack once started will increase in extent until the test specimen fails, even if the load is not increased. The cracks were similar to season cracks; the largest was generally found where the test specimen emerged from the liquid. In some cases this crack was the only one present; in others there were quite a number of cracks distributed over the length of the test specimen. Fig. 1 shows two of the test specimens, Nos. 2 and 4, after they had cracked.

In order to discover what alloys are subject to failure by cracking all the usual copper alloys will be tested.

The stresses at cracking are as follows:

KIND OF METAL.	STRESS AT CRACKING, LB. PER SQ. IN.
No. 1.—Manganese bronze, plain extruded	27 000
No. 2.—Manganese bronze, plain extruded	23 000
No. 3.—Manganese bronze, plain extruded	23 000
No. 4.—Naval brass, plain extruded	20 000
No. 5.—Muntz metal, plain extruded	23 000
No. 6.—Gun metal (88 Cu, 10 Sn, 2 Zn), cast	23 000

It appears that the stress at which cracking occurs bears some relation to the elastic limit, but this relation has not yet been definitely established. Present results indicate that the metal is liable to cracking when the stress exceeds the elastic limit by 5000 lb. per sq. in.

A detail report on the tests made is given in Table I.

Four specimens of manganese bronze have been under excessive stress for many months but have been protected from corrosion. These specimens have not cracked. Some manganese-bronze studs which were found cracked in valves in the Catskill Aqueduct were made up with red lead. One test bolt, therefore, was embedded in red-lead paste and drawn up to a stress in excess of the initial yield point. In the part of the bolt where the red lead remained liquid, the surface of the metal was stained and cracks appeared.

Two specimens of plain-extruded manganese bronze were corroded, one in ammonia, the other in a mixture of ferric chloride and hydrochloric acid, and then broken in the testing machine. The specimens gave ultimate strengths of 65,300 and 60,000 lb. per sq. in., and elongations of 43 and 42 per cent, respectively. These results, which are quite normal, indicate that corrosion does not generally injure brass when it is not under stress while being corroded.

The results thus far obtained may be summed up as follows:

1. Prolonged excessive stress does not injure the metal when it is protected from corrosion while under stress.

2. Corrosion does not generally injure the metal when not accompanied by excessive stress.

3. Corrosion accompanied by prolonged stress less than 20,000 lb. per sq. in. does not generally injure the metal.

4. Corrosion by ammonia accompanied by a prolonged stress of 20,000 lb. per sq. in. or more causes cracking similar to season cracking.

5. The foregoing results apply to manganese bronze, naval brass, Muntz metal and gun metal.

6. No copper alloy has as yet been found to which these results do not apply. It is possible, however, that such exceptions exist, either on account of higher elastic limit or greater resistance to corrosion.

CONCLUSION.

Practical experience with season cracking indicates that ammonia is not the only corrosive agent which causes cracking in overstressed copper alloys, but that the corrosion which results from the common conditions of exposure also produces cracking. Ammonia, therefore, may be regarded as a representative corrosive agent, although it attacks these alloys more quickly than the average exposure. The test with ammonia may be regarded as an accelerated test which in a few hours produces results which ordinarily would require days, weeks or months to develop, but which do not differ in kind from the results of the more common forms of exposure. Since all copper alloys in practical use are exposed to various corrosive agents it is probable that sooner or later all metal in use will be affected

by corrosion, just as much as the test specimens in the tests here reported. Therefore, it follows that the results obtained from these tests may be expected wherever copper alloys are permanently stressed over 20,000 lb. per sq. in.

It seems, therefore, that we are justified in drawing the conclusion that forgible brass and gun metal, and probably copper alloys in general, should never be stressed in practice to 20,000 lb. per sq. in. In other words, 20,000 lb. per sq. in. should be regarded as the practical ultimate strength of this group of materials. The working stress should therefore be taken as a safe fraction of this stress, say 5000 lb. per sq. in.

An important corollary of this conclusion is that the process of manufacturing rods of copper alloys must be so planned that no appreciable initial tensile stress is left in the surface of the rod, and that the proper inspection of drawn rods must include strain measurements.[1] Another important corollary is that copper alloys cannot be depended on where the stress is indeterminate, for in such cases it may or may not exceed the safe stress. These alloys, therefore, should not be used for important bolts when there is the least occasion for drawing them up very tight.

[1] For description of such measurements, see article by the writer, "Cracking of Drawn Brass," *Engineering Record*, April 11, 1914.

DISCUSSION.

THE CHAIRMAN (PRESIDENT-ELECT MANSFIELD MERRIMAN). —This very interesting paper is now open for discussion. It brings before us facts which have not been previously known, facts which indeed could scarcely have been discovered in the laboratory, but which had to be found out, first in the field and afterwards verified by laboratory methods. The Chairman.

MR. JAMES O. HANDY.—I should like to ask Mr. Jonson whether his research has extended to copper. I have seen some indication that hard-drawn copper wire in cables will crack after being corroded, while under stress. I should like to ask Mr. Jonson whether he has observed anything of that sort either in the field or in his own research. Mr. Handy.

MR. ERNST JONSON.—There is no copper specimen included in the present series, but a preliminary test made on this metal indicated that it will crack when simultaneously exposed to excessive stress and corrosion. Mr. Jonson.

MR. G. K. BURGESS AND MR. P. D. MERICA (*presented in written form*).—Mr. Jonson has undoubtedly made a distinct contribution to our knowledge of the behavior of brasses under what might be called accelerated service conditions. His work leaves little doubt but that brasses under stress are in general subject to corrosion by ammonia or ammonium hydroxide in such a way that they fail at loads much under their ultimate strengths, and indeed at loads below those at which they fail under the same stress-fatigue conditions but without corrosion. Messrs. Burgess and Merica.

There is another well-known example of the corrosion of such 60:40 brasses, namely, the corrosion by sea water of Muntz metal sheathing for ships' bottoms. Doubtless, if Mr. Jonson had used sea water instead of ammonia, he would have obtained the same kind of results; possibly, however, not in as short a time.

Of the six tests which he mentions, all but two were carried out on extruded brass, which is very possibly initially stressed, a fact which would perhaps obscure the results somewhat.

essrs. Burgess d Merica.

Most interesting are case No. 2 of manganese bronze, heated to near the melting point and quenched, and case No. 6 of gun metal, cast. It may be noted in this connection that the yield point of the latter metal ranges from 15,000 to 17,000 lb. per sq. in., whereas the specimen as corroded did not fail until 23,000 lb. per sq. in. was reached.

Mr. Jonson draws the important conclusion that any agent which corrodes brass at all will in time produce the same effect (cracking) as ammonium hydroxide, that, in other words, the "ammonia" test is a perfect acceleration test. This raises the question: why has not a great deal of brass of this sort, in the shape of castings, forgings, and rods failed in service, since some of it has undoubtedly been stressed in service up to 20,000 lb. per sq. in. while at the same time subject to corrosion by air or water or both? Data bearing on this question would do much toward deciding the validity of Mr. Jonson's conclusion.

It may be mentioned that there is one case, fairly well known, in which one type of corrosion does produce failure in the material, and others, during the course of which a greater weight of corrosion product is found, do not. Thus, the action of dilute acids (in pickling or otherwise) as well as that of alkalies (the latter according to some unpublished work of one of us) upon steel produces brittleness and cracking, whereas steel rusted in the ordinary way is still sound.

There is undoubtedly room for much investigation along these lines, and in the course of such investigation statistics should be gathered bearing on the behavior of such materials in service. From a study of these statistics, together with tests, where possible, of the materials concerned, much might be done toward settling this important question: what are the safe working loads for brasses subject to corrosion?

r. Clamer.

MR. G. H. CLAMER.—The first example of season cracking with which I have come in contact came to my notice about fifteen years ago. This was a condenser made of brass tubes of the usual composition, which had been delivered but had never been put into service. The condenser in question had been placed in the yard of one of our large railroad shops, and had stood there probably six months. During this time, the tubes had not actually corroded, but they had become what I

should have described as tarnished. In this condenser probably three quarters of the tubes cracked in such a manner that they became absolutely useless. The tubes were no doubt under stress, caused by the drawing process by which they were made, but they were not in tension, as were the bolts described by Mr. Jonson, and yet they exhibited season cracking of a very serious nature. Mr. Clamer.

Mr. Jonson.—Stress is the usual condition under which cracking occurs in all materials and under all circumstances. It is therefore highly probable that the tubes of the condenser to which Mr. Clamer refers contained heavy initial stresses put into them in the drawing. The speaker has measured such stresses in rods and has found them as high as 30,000 lb. per sq. in. at the surface. While this condenser had not been used, it had been lying in the yard for a considerable time and was therefore undoubtedly quite tarnished. The speaker has seen large quantities of rods crack in the same way before they were put into service. It is a fact that season cracking generally occurs a considerable time after the material is finished. Hence its name. This fact indicates that the stress put into the material by the cold working is not the only condition required for cracking, but that some slow change must take place in the surface of the material which gradually weakens it to a point at which the stress causes it to crack. The speaker believes that this second condition required for cracking is corrosion or tarnishing. If this corrosion is hastened by some agent such as ammonia, the cracking will occur much sooner, and if the surface is weakened by amalgamation with mercury, a crack will appear immediately, before the amalgamation has had time to proceed to any measurable depth. A brass specimen under excessive stress will break right off the very instant it is touched by a solution of mercuric nitrate. Mr. Jonson.

BATTERY ZINCS: SOME CAUSES OF DEFECTIVE SERVICE.

By Robert Job and F. F. White.

SUMMARY.

The paper states the results of an investigation of battery zincs which were of apparently satisfactory composition, but which gave decidedly different results in service.

The difference was found to be due to a segregated condition of the metal caused by the method of manufacture.

BATTERY ZINCS: SOME CAUSES OF DEFECTIVE SERVICE.

By Robert Job and F. F. White.

The serviceability of the zinc element of a gravity cell, such as is commonly used in railway service, is generally considered to depend mainly upon its composition, and it is specified usually that the percentage of iron shall be a minimum, say, not exceeding 0.10 per cent, lead not to exceed 0.50 per cent. and not less than 2 per cent of mercury.

Some years ago it was found by one of the authors that the physical condition of the zinc might have a decided effect upon its efficiency in a cell, and that it was advantageous to avoid overheating the metal and of benefit to pour into the molds at a temperature only high enough to permit the formation of solid castings.

A recent investigation which we have made has been of interest in proving the radical influence which the details of the method of manufacture exert upon the practical value of the metal in service. In the course of routine tests, two shipments of zinc were received and on analysis were found to have the following composition:

	Shipment A.	Shipment B.
Mercury, per cent	2.49	2.26
Lead, per cent	0.17	0.17
Iron, per cent	0.15	0.10

These results were within the specification limits, or nearly so, and the shipments were approved. It developed in service, however, that there was a marked difference between the quality of the two lots, and that shipment A was far superior to shipment B. The zincs in the latter lot, after comparatively short service, became coated with copper which protected the zinc from the action of the electrolyte, and in consequence the cell became "dead" when but little of the zinc had been dissolved.

In view of the satisfactory average analysis of the zincs, it was felt that the difference in service was probably due to

characteristics other than composition, and an investigation was made of each of the components of the cell.

The copper sulfate contained 0.75 per cent of iron, figured as the crystallized sulfate ($FeSO_4.7H_2O$). This amount was the maximum permitted by the specifications, but the same copper sulfate gave good results when used with zincs of shipment A. It was decided then that the difference in the zincs was probably physical, and in order to develop possible variations in structure

(*a*) Good. (*b*) Defective.

FIG. 1.—Transverse Sections of Zinc Plates (× 2).

transverse sections were taken from several samples in each shipment, and were polished and etched lightly with a solution of ammonium sulfide, acidified with hydrochloric acid. The sections are shown in Fig. 1 (*a*) and (*b*).

Fig. 1 (*a*) represents the good zincs and Fig. 1 (*b*) the defective shipment, at a magnification of two diameters in each case. The etchings and subsequent microscopic examination showed marked difference. The samples represented by Fig. 1 (*a*) were fine grained and of uniform structure, clear to the outside surface

of the zincs, whereas those of Fig. 1 (*b*) were coarse grained, indicating pouring into molds at a high temperature. Also, a distinct band of bright metal was seen around the contour of Fig. 1 (*b*). The natural inference was that the outside metal contained little mercury, and in order to determine this we took borings from each section around the surface to a depth of $\frac{1}{16}$ in., and found the mercury content for Fig. 1 (*a*), representing shipment A, to be 2.49 per cent, that for Fig. 1 (*b*), representing shipment B, 0.64 per cent. These results showed the condition clearly and indicated the main cause of failure of shipment B.

It is a well-known fact that mercury volatilizes to a considerable extent when added to molten zinc, unless suitable precautions are taken; consequently it is necessary not only to avoid overheating the bath of metal, but also to keep the molds cooled. Evidently in the case of shipment B this latter practice had not been followed, and hence the proportion of mercury upon the surface of the zincs was extremely low and evidently insufficient to protect the zincs from local action.

The foundry practice necessary to produce well-mixed, sound, and serviceable battery zincs is not difficult, but it is particularly essential that overheating of the metal be avoided, either in the melting pot or in the mold, and that after addition of the mercury the bath be kept carefully stirred in order to avoid segregation.

In onclusion, acknowledgment is made of the assistance of Mr. H. W. Lewis, Signal Engineer of the Lehigh Valley Railroad Co., in noting the characteristics of the shipments in service, and for help rendered in carrying out the investigation.

DISCUSSION.

Mr. Harrington. MR. C. O. HARRINGTON, JR.—I notice Mr. Job agrees that it is impossible to maintain the same percentage of mercury near the periphery of the zinc. There is no statement made as to how close the percentage of mercury at the periphery, or within $\frac{1}{16}$ in. of the periphery, should be kept as compared with the percentage of mercury in the body of the zinc. Did Mr. Job's investigations show what proportion of mercury we might expect at the periphery of the zinc? Another point: does he not think it would be advisable to inspect the gravity battery zincs by taking a turning of about $\frac{1}{16}$ in. in depth from the periphery, analyzing that turning, and comparing it with a drilling from the body of the zinc and specifying that the variation in the percentage of mercury should fall within certain limits, so that there should not be more than a certain variation between the body and the periphery?

Mr. Job. MR. ROBERT JOB.—In regard to the first question, the appearance of the photograph will, perhaps, give a very good indication and I have given in the paper the percentage of mercury in borings taken to the depth of $\frac{1}{16}$ in. In shipments of the good zincs we have found practically the same proportion of mercury as was found in the test zinc. We went to a depth of $\frac{1}{16}$ in. so that we might determine about what difference might be expected with good foundry practice. If we were to reduce the distance, say to $\frac{1}{64}$ in., naturally we should find a considerably greater difference, and theoretically it is almost impossible to get exactly the same percentage of mercury on the very outside of the zinc as at the center of the casting. For practical purposes we find, however, that the percentage of mercury within $\frac{1}{16}$ in. should be reasonably close to the percentage found in the main body of the zinc in order to avoid difficulties in service due to segregation. Also, as mentioned in our paper, it is important that the casting shall have a fine granular structure, and about two per cent of mercury is sufficient when the composition in other respects is normal.

The point of particular interest in connection with these shipments is that shipment B gave practically no service and was a failure, whereas shipment A, which contained practically the same amount of mercury and whose composition in other respects was essentially the same as shipment B, gave excellent service. Mr. Job.

MR. JESSE L. JONES.—I recall that Mr. Joseph Richards, the father of Professor Richards of Lehigh University, used to run down battery zincs and recover the mercury from them. His process was simply to melt the battery zincs at night in an iron kettle over which was placed a sheet-iron hood with a trough at its base. In the morning the mercury would be found condensed in the trough. I do not know whether he recovered all the mercury in the battery zincs in this way, but he did recover a large portion of it. Mr. Jones.

MR. O. LINDER.—I had occasion some years ago to carry on an investigation of battery zincs and found at that time that the most prominent impurities in the zinc were not iron and lead, but cadmium. The cadmium present in the zinc is apt to run up to a high percentage; I think I have seen as much as 4 or 5 per cent. This element is generally overlooked in the analysis of battery zinc; consequently in making investigations of that subject it is very important that we have not only the iron and lead and other impurities to consider, but also particularly the cadmium. There is no question but what the distribution of the percentage of mercury has considerable effect upon the serviceability of zinc, but I have always found that a small trace of mercury would serve the purpose while even a small amount of cadmium, say about 0.5 per cent, would offset any good effect a considerable amount of mercury might have. Mr. Linder.

MR. JOB.—In reply to the last speaker, I may say that careful examination was made for cadmium, and that practically no cadmium was present in either sample. There was practically no difference in the amount present in each, and in both cases it was not more than a bare trace, much less than 0.1 per cent. Mr. Job.

MR. HARRINGTON.—I should like to ask Mr. Job whether the effect of a $\frac{1}{64}$-in. coating of incorrect mercury percentage deserves to be considered in zincs. Would not the effect disappear very shortly as the surface of the zinc dissolved? Mr. Harrington.

Mr. Job. MR. JOB.—The main trouble was that as soon as the zinc was placed in service it became coated with copper, and did not "clean itself." Therefore, practically no action took place subsequently, and as a consequence the zinc became useless in a very short time.

THE DETERMINATION OF SPELTER COATING ON SHEETS AND WIRE.

By J. A. Aupperle.

SUMMARY

There are several methods used for the determination of the weight of spelter coating. The lead-acetate method has been used where great accuracy is desired, while the Preece copper-sulfate dip test has been used to a very large extent by both the manufacturer and purchaser as a basis of specification. This refers more particularly to wire than to galvanized sheets. The Preece test gives an approximate idea of the amount of coating present. The results obtained vary somewhat with the temperature and manipulation.

The proposed method for determining the weight of spelter coating is one that yields extremely accurate results, and a determination can be made in less time than is required for the average Preece test. The method consists essentially of weighing the samples, preferably on a balance provided with rider weights, the section weighed being of such dimensions that the number of grams of coating corresponds to ounces of coating per square foot. The samples are immersed in a concentrated solution of hydrochloric acid containing antimony chloride. This constituent accelerates the solution of the spelter coating and by superficially plating the base metal prevents the action of the hydrochloric acid thereon.

It is believed to be more desirable to state the amount of coating in ounces per square foot. In the case of wires this refers to actual surface, while in galvanized sheets it refers to one surface only. In making direct comparisons, therefore, between the weight of coating on wire and on galvanized sheets the figure obtained on wire should be doubled.

THE DETERMINATION OF SPELTER COATING ON SHEETS AND WIRE.

By J. A. Aupperle.

For many years the Preece copper-sulfate test has been used to determine the amount of galvanized coating on sheets and wire. Committee A-5 on the Corrosion of Iron and Steel reported to the Society in 1911 on this test as follows:

"It is, however, the unanimous opinion of the committee that the well-known Preece copper-sulfate test is unreliable and should be abandoned entirely as a basis of specification with respect to galvanized sheet and plate. In respect to wire, the Preece test has the advantage of being quick and simple, and if carried out in the proper manner yields comparative results of value. In the opinion of the committee, the lead-acetate test is preferable to the copper-sulfate test for determining or specifying the weight of zinc coatings."

The lead-acetate method recommended by Committee A-5 in 1911 yields very accurate and satisfactory results, but the length of time required for making the test seriously limits the scope of its usefulness. The results obtained with the method described in this paper compare very favorably with those of the lead-acetate method.

There is much to be desired in the method of expressing the weight of coating on wire products in order to have an intelligent understanding as to the weight of coating per unit area. It has been customary to express the weight of coating on wire in pounds per mile, while on sheet products the results are usually expressed in ounces per square foot. Obviously, the coating on wire expressed in pounds per mile would have a different meaning for each gage of wire. If the results are expressed in ounces per square foot of surface on both wire and sheets, there will be a better understanding as to the thickness of coating on the respective products. In stating the weight of coating on galvanized sheets it is customary to express the weight based on one surface only, that is, a sheet containing 2 oz. of coating per square foot really contains 1 oz. on each side of the sheet.

It is proposed to express the weight of coating on wire in ounces per square foot, and also to use such lengths of wire that the number of grams of coating found will be equivalent to ounces per square foot, without calculation. These lengths must be such that the surface coated is equal to 5.079 sq. in. It is likewise proposed that the samples for determining the weight of coating on galvanized sheets shall be $2\frac{1}{4}$ by $2\frac{1}{4}$ in. (area = 5.079 sq. in.). The number of grams of coating on a section of this size will also express the weight of coating in ounces per square foot without calculation.

The method for determining the weight of spelter coating consists of using a small amount of antimony chloride in hydrochloric acid (sp. gr. 1.20). Antimony chloride appears to hasten the solution of the coating, and after the coating has dissolved a thin film of antimony plates on the surface of the base metal and retards the solution of iron or steel. Experiments have shown that sheet steel $2\frac{1}{4}$ by $2\frac{1}{4}$ in. which loses 50 mg. in five minutes in cold hydrochloric acid (sp. gr. 1.20), will lose in that time only 1 mg. in the same acid containing 80 mg. of antimony per 105 cc. of acid.

In the proposed method the metal is immersed in the acid only one minute, which is long enough to dissolve several grams of coating, yet the amount of iron or steel dissolved is negligible. The small amount of antimony that plates on the surface of the sample can easily be removed by scrubbing under running water. This method is one of the most rapid and accurate with which the writer is familiar, and a determination can be made in less time than is occupied in making the Preece test.

Sheets.—For determining the weight of coating on galvanized sheets cut several samples $2\frac{1}{4}$ by $2\frac{1}{4}$ in. from various parts of the sheet. These samples, about five in number, should be weighed together and immersed singly for one minute in 100 cc. of hydrochloric acid (sp. gr. 1.20), to which has been added 5 cc. of antimony chloride prepared by dissolving 20 g. of antimony trioxide in 1000 cc. of hydrochloric acid (sp. gr. 1.20). The same 100 cc. of hydrochloric acid can be used for at least five samples. Five cubic centimeters of the antimony chloride, however, should be added for each sample on account of the antimony being removed from the solution by the iron.

The samples are washed and scrubbed under running water, dried with a towel, and laid in a warm place for a few seconds. The samples are again weighed together and the number of grams lost is divided by the number of samples taken. Each gram corresponds to 1 oz. of coating per square foot.

TABLE I.—LENGTHS OF WIRE TO GIVE GRAMS OF COATING EQUIVALENT TO OUNCES PER SQUARE FOOT.

Gage No.	Diameter, in.	Length for Test,	
		in.	cm.
0	0.340	$4\frac{2}{16}$	12.1
1	0.300	$5\frac{6}{16}$	13.7
2	0.284	$5\frac{11}{16}$	14.5
3	0.259	$6\frac{4}{16}$	15.9
4	0.238	$6\frac{13}{16}$	17.3
5	0.220	$7\frac{6}{16}$	18.7
6	0.203	$7\frac{15}{16}$	20.2
7	0.180	9	22.8
8	0.165	$9\frac{13}{16}$	24.9
9	0.148	$10\frac{15}{16}$	27.7
10	0.134	$12\frac{1}{16}$	30.6
11	0.120	$13\frac{8}{16}$	34.2
12	0.109	$14\frac{13}{16}$	37.7
13	0.095	17	43.2
14	0.083	$19\frac{8}{16}$	49.5
15	0.072	$22\frac{7}{16}$	57.0
16	0.065	$24\frac{14}{16}$	63.2
17	0.058	$27\frac{14}{16}$	70.8
18	0.049	33	83.8

Wire.—A small section of the galvanized wire should be stripped in hydrochloric acid containing antimony chloride. The diameter of the black wire should then be carefully measured in order to determine the length of wire, such that the number of grams of coating will represent the number of ounces per square foot of surface. These lengths are given in Table I. In the lighter wires, however, it will be found convenient to use some fraction of these lengths.

The method of making the test is very similar to that outlined for galvanized sheets, except that the wire is first cleaned with carbon tetrachloride or gasoline, and after being carefully weighed is placed in a tall glass cylinder containing hydrochloric acid (sp. gr. 1.20), to which has been added from 2 to 3 cc. of antimony-chloride solution of the same strength as used on galvanized sheets. The reason for using one-half the amount of antimony chloride in the case of wire is on account of taking one-half the area. As previously stated, the coating on galvanized sheets is expressed in ounces per square foot, considering one side only, when in reality this amount of coating represents two square feet of surface. After immersing the entire length of wire for one minute it will be found convenient to pour the acid solution into another tall cylinder in order to facilitate removing the wire. The wire is then scrubbed under running water, wiped, thoroughly dried in a warm place for a few seconds and again weighed. Each gram lost corresponds to 1 oz. of coating per square foot. For direct comparison with the weight of coating as expressed on galvanized sheets, this figure should be doubled.

DISCUSSION.

Mr. Harrington. MR. C. O. HARRINGTON, JR.—I understand that the copper-sulfate test is sometimes used for determining the thickness of sherardized iron plates, sherardizing being, I believe, a zinc coating formed by applying zinc oxide to the metal in an oven at nearly the melting point of zinc. Now the copper-sulfate test will apparently serve as an indicator of the thickness of the sherardized coating on steel or iron. Do you think the proposed test would be a preferable means of determining the thickness of the sherardized coating on iron rods, or have you had any experience which would lead you to draw such a conclusion?

Mr. Aupperle. MR. J. A. AUPPERLE.—We have made some experiments with this method on sherardized metals and have obtained very satisfactory results. It is generally assumed that the Preece copper-sulfate test is not very reliable and not generally used for sherardized products.

Mr. Walker. MR. PERCY H. WALKER.—I should like to ask about the details of carrying out this test. It is not entirely clear to me whether the author adds all of the antimony solution, that is, 25 cc., to the 100 cc. of acid, before beginning dipping the five pieces of sheet metal, or whether he adds 5 cc. and dips one sheet and then adds five more, etc.

Mr. Aupperle. MR. AUPPERLE.—The test is made by adding 5 cc. of antimony chloride just previous to making the test on each individual sample.

Mr. Jones. MR. JESSE L. JONES.—The method given by Mr. Aupperle is a very good one where the spelter coating is to be determined on sheets and wire only; but miscellaneous articles do not lend themselves to this method as well as sheets and wire for the reason that they cannot be weighed conveniently and accurately and the surface area cannot be readily determined. For this reason I think Mr. Aupperle's method and other methods that

have been recommended from time to time as substitutes for the copper-sulfate method leave much to be desired. Mr. Jones.

MR. AUPPERLE. It is true that the irregular shape of some of the galvanized products would make the method inapplicable, and for that reason, it is limited to the determination of spelter coating on sheets and wire. Mr. Aupperle.

THE EFFECT OF FINER GRINDING AND A HIGHER SO_3 CONTENT UPON THE PHYSICAL PROPERTIES OF PORTLAND CEMENT.[1]

By P. H. Bates.

SUMMARY.

The question of the finer grinding and the addition of more SO_3 to Portland cement is frequently discussed, and the consensus of opinion seems to be that further investigation is needed. In the present paper ten commercial cements have had more SO_3 added to them, have been ground finer, and have been both ground finer and had more SO_3 added. From the four groups of ten cements each, the customary physical tests and small specimens have been made. In addition cylinders of $1:1\frac{1}{2}:4\frac{1}{2}$ concrete have been made, and expansion bars of neat and 1:3 standard sand mortars. Some of the neat tension briquettes have also been examined microscopically for relative amounts of hydration.

The results show that the time of set is affected somewhat by each of the above treatments, finer grinding tending to produce a quicker set, and the addition of more SO_3 a quicker initial but slower final set. The addition of SO_3 to the coarser-ground cements does not materially affect the strength; finer grinding produces considerable increase; while the addition of SO_3 to the finer-ground cements tends to produce results very slightly less than those obtained when they contain the normal amount. Expansion measurements show that the addition of SO_3 to the coarse cements produces a large increase in length of neat cements; to finer-ground cements the increase is not so great. Finer grinding alone does not materially affect the expan-

[1] By permission of the Director of the Bureau of Standards.

sion due to hydration; the expansion of the mortar bars is not materially affected by the use of the different cements.

All conclusions made in this paper are deduced from results obtained from specimens tested at the end of 90 days. Specimens have been made to be tested at the end of six months, one year, and two later periods, and consequently the present conclusions may have to be materially modified. The results of the 6-month specimens will be presented at the coming meeting of the Society.

THE EFFECT OF FINER GRINDING AND A HIGHER SO_3 CONTENT UPON THE PHYSICAL PROPERTIES OF PORTLAND CEMENT.

By P. H. Bates.

Introductory.

That there is a desire for more information in regard to the finer grinding and the further addition of SO_3 to Portland cement than that now required or allowed by specifications, is shown by the fact that the International Association for Testing Materials has appointed two committees for investigating these subjects (Problems Nos. 30 and 55).

That finer grinding of the clinker—so that an amount greater than 75 per cent passes through a 200-mesh sieve—gives more desirable results, when measured by the strength developed in mortar or concrete, is acknowledged by both producer and consumer; both, however, are somewhat in doubt whether the results justify the extra cost of the cement which will necessarily result from the finer grinding. If the increased fine material will produce strengths which will justify a reduction in the amount of cement used, then the matter of cost may become negligible and finer grinding may be justified.

The question of SO_3 is one which would seem to interest the producer most, and to interest some producers more than others. The composition of the cement, the climatic conditions under which the cement is used, and the amount of finely-ground particles play important parts in the SO_3 requirements. According to the generally accepted conclusions, high-alumina cements require more SO_3 to make them usable during the hot humid seasons than the low-alumina and high-iron-oxide cements; and further, the finer a cement is ground the more SO_3 is required to prevent it from setting too quickly. Yet the consumer is inclined to consider this addition of SO_3 somewhat in the nature of an adulterant, or at least a necessary evil; and

is inclined to desire to keep the permissible amount down to a minimum, without regard to the connection of composition and degree of fineness with SO_3 requirements.

By taking a number of cements and determining their physical properties and the strengths which they developed in neat, mortar and concrete specimens, and comparing these results with those obtained upon the same cements (1) when ground so that about 12 per cent more passed a 200-mesh sieve, (2) when the same cements had the SO_3 content increased to about $2\frac{1}{2}$ per cent, and (3) when the same cements had the SO_3 content increased to this greater amount and were further ground to the above-mentioned fineness, it was thought some interesting and valuable data on these subjects would be obtained.

The following brands of cement were used in this investigation: Atlas (Lehigh District), Vulcanite, Crescent, Ironton, Ajax, Medusa White, Alpha (West Virginia), Superior, Lehigh (Western Pennsylvania) and Universal (Universal, Pa.). These are hereafter referred to by letter without any consideration of the above-given sequence. These are not only representative brands but they also give a wide variation in composition and represent several varieties of raw materials.

PREPARATION OF MATERIAL AND TEST SPECIMENS.

On receipt of the various cements at the laboratory, the entire sample was mixed and then divided into four parts. The following tests and specimens were immediately made with one of these parts: fineness, specific gravity, time of set, constancy of volume, chemical analysis, neat tension and compression test specimens, 1:3 standard sand mortar tension and compression test specimens, $1:1\frac{1}{2}:4\frac{1}{2}$ concrete specimens (6 by 12 in. cylinders), neat and 1:3 standard sand mortar expansion test specimens (1 sq. in. in cross section by 13 in. long with 10-in. gage length), and a set of neat and 1:3 standard sand mortar specimens (both tension and compression) aged partly in water and partly in air according to the method recommended by the German cement specifications. The tests with this part of the cement are referred to in the text by the numeral "1" in connection with the proper letter.

Another part of the original sample was then placed in a ball

mill, lined with porcelain and containing flint balls, and ground to the fineness shown in Table I. This mill is of the "intermittent" type, and the entire charge was constantly subjected to

TABLE I.—CHEMICAL ANALYSIS, FINENESS, AND SPECIFIC GRAVITY OF CEMENTS.

Cement No.	Percentages of								Loss on Ignition, per cent.	Fineness, per cent passing		Specific Gravity.	Grinding Mills.
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O		100-mesh.	200-mesh.		
A-1	19.53	8.42	2.95	62.12	1.63	1.47	0.02	0.23	3.75	96.6	79.2	3.021	Kent
A-2	....	....	....	....	....	2.46	....	....	....	....	....	3.016	and
A-3	....	....	....	....	....	....	....	....	....	99.4	88.8	3.032	Tube
A-4	....	....	....	....	....	2.52	....	....	....	99.6	91.6	3.032	Mills.
B-1	20.43	7.55	2.52	62.76	2.89	1.49	0.32	0.82	1.26	95.8	79.4	3.124	Komi-
B-2	....	....	....	....	....	2.40	....	....	....	....	....	3.108	nuter &
B-3	....	....	....	....	....	....	....	....	....	99.2	89.2	3.121	Tube
B-4	....	....	....	....	....	2.39	....	....	....	99.2	89.8	3.121	Mills.
C-1	21.78	7.07	3.83	62.52	1.44	1.42	0.03	0.67	1.21	98.8	75.4	3.153	Maxecon
C-2	....	....	....	....	....	2.40	....	....	....	....	....	3.134	and
C-3	....	....	....	....	....	....	....	....	....	99.2	86.8	3.151	Tube
C-4	....	....	....	....	....	2.39	....	....	....	99.4	88.6	3.135	Mills.
D-1	23.32	6.49	2.59	60.34	2.17	1.65	0.59	0.70	2.43	94.8	77.8	3.122	Lehigh
D-2	....	....	....	....	....	2.43	....	....	....	....	....	3.115	Fuller
D-3	....	....	....	....	....	....	....	....	....	99.4	88.6	3.124	Mills.
D-4	....	....	....	....	....	2.47	....	....	....	99.4	90.0	3.111	
E-1	22.82	6.27	2.09	62.79	2.76	1.41	Trace	0.63	1.46	95.0	75.8	3.147	
E-2	....	....	....	....	....	2.44	....	....	....	....	....	3.123	Griffin
E-3	....	....	....	....	....	....	....	....	....	99.6	89.8	3.156	Mills.
E-4	....	....	....	....	....	2.47	....	....	....	99.6	91.0	3.143	
F-1	22.70	6.18	2.27	62.30	3.25	1.55	0.06	0.47	1.35	93.8	79.0	3.153	
F-2	....	....	....	....	....	2.38	....	....	....	....	....	3.137	Tube
F-3	....	....	....	....	....	....	....	....	....	99.4	90.8	3.155	Mills.
F-4	....	....	....	....	....	2.53	....	....	....	99.3	90.6	3.141	
G-1	21.35	6.91	2.05	62.56	2.54	1.61	0.02	0.76	2.50	96.4	82.2	3.086	Ring
G-2	....	....	....	....	....	2.34	....	....	....	....	....	3.077	Rolls &
G-3	....	....	....	....	....	....	....	....	....	99.8	91.2	3.103	Tube
G-4	....	....	....	....	....	2.50	....	....	....	99.6	91.0	3.073	Mills.
H-1	20.79	7.14	2.61	60.34	3.23	1.64	0.40	0.75	3.17	95.6	77.6	3.114	Hunt-
H-2	....	....	....	....	....	2.47	....	....	....	....	....	3.114	ington &
H-3	....	....	....	....	....	....	....	....	....	99.6	91.0	3.115	Tube
H-4	....	....	....	....	....	2.46	....	....	....	99.0	90.0	3.118	Mills.
J-1	20.38	7.56	5.05	61.96	0.88	1.60	0.07	0.75	1.55	96.4	81.4	3.138	Ring
J-2	....	....	....	....	....	2.47	....	....	....	....	....	3.142	Rolls &
J-3	....	....	....	....	....	....	....	....	....	99.2	92.7	3.149	Tube
J-4	....	....	....	....	....	2.50	....	....	....	99.7	93.6	3.142	Mills.
K-1	25.12	5.38	0.49	64.71	1.40	1.20	0.04	0.56	1.36	98.4	81.8	3.118	Kent
K-2	....	....	....	....	....	2.41	....	....	....	....	....	3.108	and
K-3	....	....	....	....	....	....	....	....	....	100.0	92.4	3.122	Tube
K-4	....	....	....	....	....	2.34	....	....	....	99.8	92.2	3.113	Mills.

grinding. The tests and specimens enumerated above were then made with this portion, and are referred to by the numeral "3" in connection with the proper letter.

A third part was placed in the same ball mill and sufficient plaster added to make the total amount about 2.5 per cent and then ground until about the same fineness as the second part, after which the various test specimens were made and are referred to by the numeral "4."

About 50 lb. of the last part were placed in the mill with the same amount of plaster as used with the third portion. This was ground for about 15 minutes with but a small quantity of balls, so that the action was more of a mixing than a grinding. Then the remainder of this portion was placed in the mill and the mixing continued for 10 minutes; after which the various tests and specimens were made and are referred to by the numeral "2."

The chemical analyses of the original samples, the percentage of SO_3 in the treated samples, the fineness of the samples, together with the kind of grinding machines used in the cement plants, are given in Table I.

TIME OF SET.

The time of set as determined by both the Vicat and Gilmore needles is shown in Table II, which also gives the normal consistency as determined by the ball method, as well as the penetration of the Vicat plunger when the consistency as determined by the ball method was used.

The tendency of the increasing of the SO_3 content has been to make the initial set invariably quicker, both in the case of the samples as received and as re-ground. This tendency is much more marked with the Vicat test specimens than with the Gilmore specimens, and when the former specimens showed a very quick initial set they also showed a decided evolution of heat, which was not the case with the Gilmore specimens. This characteristic of the Vicat specimens, to give a much quicker set than the Gilmore specimens in the case of cements which are inclined to be somewhat quick setting, is well known, and is undoubtedly due to the fact that such cements evolve heat during the period of initial set. This heat is conserved in the former specimen, which is made of a larger mass and rather well insulated, but more quickly dissipated in the smaller non-insulated Gilmore specimen. The finer grinding has also decreased the time of

the initial set, though not to the same degree as has been produced by the additional SO_3.

The final set has been made decidedly slower by the addition

TABLE II.—TIME OF SET AND NORMAL CONSISTENCY OF CEMENTS.

Cement No.	Time of Set.				Percentage of Mixing Water for Normal Consistency.	Penetration of Vicat Plunger, mm.
	Initial Set, hr. and min.		Final Set, hr. and min.			
	Vicat Method.	Gilmore Method.	Vicat Method.	Gilmore Method.		
A-1	2 : 15	3 : 55	5 : 35	6 : 40	23.0	12
A-2	0 : 18	3 : 15	6 : 20	10 : 15	23.5	10
A-3	1 : 00	3 : 00	4 : 25	5 : 35	23.0	10
A-4	0 : 08	2 : 15	5 : 25	6 : 50	23.5	9
B-1	3 : 00	4 : 40	6 : 10	6 : 40	22.5	7
B-2	2 : 45	4 : 45	6 : 55	9 : 40	23.0	7
B-3	0 : 25	2 : 00	5 : 25	5 : 55	24.0	7
B-4	3 : 00	4 : 20	6 : 00	7 : 10	24.0	6
C-1	3 : 40	4 : 55	7 : 00	7 : 40	21.0	10
C-2	1 : 50	4 : 00	7 : 10	11 : 10	21.5	8
C-3	3 : 30	4 : 35	5 : 50	7 : 00	21.5	7
C-4	1 : 45	3 : 50	6 : 35	7 : 15	22.0	10
D-1	3 : 00	4 : 30	8 : 30	9 : 45	21.0	8
D-2	1 : 35	3 : 20	8 : 45	10 : 00	22.0	10
D-3	2 : 15	3 : 25	8 : 40	9 : 35	22.0	9
D-4	1 : 50	2 : 40	9 : 10	10 : 25	22.0	11
E-1	4 : 05	4 : 35	9 : 20	10 : 05	21.0	7
E-2	0 : 45	3 : 00	9 : 50	10 : 50	22.0	7
E-3	3 : 10	3 : 35	8 : 45	10 : 00	22.5	12
E-4	0 : 15	2 : 40	9 : 00	10 : 10	22.5	11
F-1	4 : 05	5 : 25	9 : 25	10 : 45	21.5	7
F-2	2 : 20	5 : 15	10 : 20	11 : 00	22.5	7
F-3	4 : 00	5 : 00	8 : 30	9 : 40	22.5	10
F-4	2 : 35	4 : 15	6 : 45	9 : 45	22.5	7
G-1	3 : 35	6 : 00	12 : 00	12 : 45	22.5	13
G-2	4 : 40	5 : 45	13 : 00	13 : 00	23.5	6
G-3	3 : 15	5 : 10	11 : 15	12 : 00	23.0	11
G-4	3 : 10	5 : 05	10 : 30	13 : 00	23.0	7
H-1	3 : 45	5 : 10	8 : 00	10 : 45	21.5	9
H-2	2 : 40	5 : 00	11 : 20	13 : 10	21.5	7
H-3	3 : 05	3 : 50	5 : 30	6 : 00	22.0	6
H-4	3 : 00	3 : 45	6 : 45	12 : 10	22.0	6
J-1	3 : 40	3 : 55	9 : 50	10 : 55	22.0	10
J-2	3 : 30	5 : 20	9 : 10	10 : 50	22.5	7
J-3	3 : 05	4 : 45	7 : 30	8 : 45	23.0	7
J-4	2 : 10	3 : 40	8 : 00	9 : 00	23.0	9
K-1	4 : 20	5 : 10	10 : 05	11 : 35	21.0	..
K-2	3 : 35	4 : 20	7 : 05	8 : 20	21.0	..
K-3	2 : 25	3 : 45	5 : 25	6 : 15	21.0	8
K-4	2 : 35	4 : 00	5 : 25	6 : 15	21.0	7

of the SO_3—just the opposite of the result shown by the initial set—whereas, the finer grinding has made it usually quicker. The latter result is what would be expected; the former appears

anomalous when considered in connection with the initial set results. While but one determination is shown in Table II for each cement, yet always two, and in the majority of cases three or four determinations were made, and all showed the same tendency. These were all well-aged cements when mixed, and that further SO_3 should have increased the final set to a time beyond that allowed by specifications needs no comment other than that possibly cements with increased SO_3 content could not be stored as long as those with normal SO_3 content without danger of being rejected for too slow final set.

As mentioned before, the SO_3 was added in the form of plaster of paris and not as gypsum, and consequently did not contain any water of hydration which would be evolved in the process of grinding and so hydrate the more readily hydrating constituents of the cement. This was done purposely, since the investigation is concerned with addition of SO_3, and not with the addition of SO_3 plus water. The latter would have added considerably to the study, since cements would have had to be included to which water, SO_3, and SO_3 and water had been added.

However, the addition of SO_3 has produced but three cements which show too quick an initial set, according to specifications, and then only according to the Vicat method. The fine grinding has produced the same effect in one case. The Gilmore specimens all give results well within the specification limits, and consequently it may be stated that either treatment of the original cements has not produced any undesirable results from the standpoint of initial set. In the case of final set, the additional SO_3 has increased the time beyond the specification limits in rather a large number of cases, but with fresher cements these increases would hardly have been sufficient to exceed these limits; and at the same time the additions would have secured that for which some manufacturers are particularly anxious, a slower final set of cements during the hot seasons.

Strengths Developed.

The strengths of all specimens are shown in Table III. These are the averages of three specimens in every case.

Neat Specimens.—As usual the neat specimens showed more variations in the results than any of the others; this was partic-

ularly true of the tension briquettes. But the statement[1] frequently made that the finer-ground cements do not produce a neat specimen having a higher breaking strength than the coarser-ground cements, is not borne out by this investigation. That the cements cited in these references should have produced low neat strengths is not surprising, since all were quick setting, and in the reground condition gave initial sets of from 1 to 4 minutes and final sets of 4 minutes to 1 hour. There would be considerable trouble in molding such cements before the initial set was passed and in not destroying some of the strength developed by the quick-setting constituents.

The general deductions to be made, however, are that the grinding has produced an increased strength which even becomes more marked when the results of the specimens made from the finer-ground material plus the additional plaster are examined. Furthermore, the addition of the plaster without any regrinding has generally increased the strength over that developed by the cement as received.

So far as retrogression of strength is concerned, this phenomenon has not been shown by the compression specimens; with the tensile specimens it is quite pronounced, and is possibly more pronounced with the specimens containing the additional plaster. This is not shown however, until after the 28-day period in the case of the coarser-ground cement, while with the specimens from the finer-ground cement it appears usually between the 7 and 28-day periods.

Mortar Specimens.—This class of specimens, both in tension and compression, shows the value of the grinding, and particularly the grinding plus the additional plaster, over those made from the other samples of cement. It is further noticed that the addition of the plaster without grinding has a tendency to reduce the strength of these specimens, particularly those broken in compression. Thus at the end of the 28-day period there are five of the cements, and at the end of 90 days six, thus treated, which show a low strength in comparison with the untreated. It might consequently be assumed that this disruptive action is still continuing, gradually overcoming the cohesive strength, and that

[1] Taylor, "Practical Cement Testing," p. 106; Butler, "Portland Cement," p. 206.

in time it will become more marked. Later tests will confirm or disprove this theory. In the case of the finer-ground cements to which more SO_3 was added, it is possible that the additional early strength gained by finer grinding has been sufficient to more than counterbalance the disruptive action of the SO_3. If this is due to a crystallin growth of sulfo-aluminate of lime, which would tend to cause a disruption of the specimen, it would surely be more pronounced with the tension test specimens, whereas such is not the case, the addition of SO_3 not having produced a marked decrease in strength in this case.

Combined Aging Specimens.—Neat and 1:3 standard sand mortar specimens, for breaking in both tension and compression, were made to be tested at the end of 28, 90, 180 and 365 days. The 28-day specimens were stored as usual for the first 24 hours in the damp closet, then in water for 6 days, and then in air for the remaining time. In the case of those to be tested at the other periods, the same ratio between the time stored in water and in air was maintained. Storing the 28-day test specimens in this manner is the test so much preferred in Germany on account of its simulating more nearly the aging which the greater part of all concrete structures receives.

The results obtained with the early specimens have been most striking, particularly on comparing the low strengths obtained with the neat tension test briquettes with the very high strengths of the sand tension test briquettes, many of the latter breaking higher than the neat specimens.

The neat compression test specimens do not show the low results of the neat tension test specimens, and the strengths are not relatively so high as those of the sand compression specimens, the latter in a number of cases being higher than the similar 90-day normally stored ones. But these are results which are comparatively well known.[1]

That the neat specimens made from the finer-ground cements, when broken in tension, should show such low results is difficult to explain. If they have not been stored in water sufficiently long to secure the extra hydration, which they needed on account

[1] Taylor, "Practical Cement Testing," p. 132; Kleinbogel, "Old and New Methods of Storing Specimens under German Cement Specifications," *Tonind. Zeit.*, Jan. 20, 1912.

TABLE III.—STRENGTHS DEVELOPED BY VARIOUS SPECIMENS.

ALL VALUES, WHICH ARE IN POUNDS PER SQUARE INCH, ARE THE AVERAGE OF THREE SPECIMENS.

Kind of Specimen.		Tested at end of	Cement No. A 1	A 2	A 3	A 4	Cement No. B 1	B 2	B 3	B 4	Cement No. C 1	C 2	C 3	C 4	Cement No. D 1	D 2	D 3	D 4	Cement No. E 1	E 2	E 3	E 4
Stored in Water	Neat Tension	24 hours	232	257	272	445	205	442	347	409	430	534	349	515	377	442	367	481	346	259	440	399
		7 days	645	639	667	844	720	840	882	863	629	769	847	927	662	679	574	818	773	875	978	864
		28 "	708	883	670	736	625	900	697	887	833	988	861	941	727	738	716	823	839	875	821	878
		90 "	650	668	691	655	702	745	720	618	677	876	884	778	712	779	695	722	803	757	927	1 073
		180 "	660	798	621	677	652	767	622	775	752	895	715	807	712	727	657	757	844	818	819	864
	Neat Compression	7 days	6 332	6 830	8 043	9 159	7 137	7 703	7 109	7 798	8 013	9 035	8 705	9 690	7 394	7 562	8 392	8 316	8 109	8 198	11 046	11 284
		28 "	9 825	10 372	12 050	11 825	8 478	8 567	8 149	9 942	10 397	12 068	9 539	12 018	9 237	10 484	11 408	10 077	12 362	12 568	14 853	14 379
		90 "	9 938	11 617	14 617	17 914	10 952	10 764	10 619	14 082	12 359	14 748	13 808	11 487	14 263	12 342	14 015	13 954	13 873	14 968	17 644	18 020
		180 "	8 721	10 712	11 862	11 008	9 296	10 687	9 317	10 292	10 429	11 979	10 421	11 804	11 890	13 051	13 508	14 552	17 728	16 168	17 408	20 170
	Sand Tension	7 days	196	246	251	338	314	351	325	408	274	329	296	411	235	268	221	305	300	376	306	268
		28 "	312	381	344	431	394	381	422	481	316	398	392	424	329	384	400	473	427	412	455	453
		90 "	343	425	373	445	417	463	450	483	414	405	458	497	407	461	474	498	430	401	562	506
		180 "	327	402	399	462	462	425	463	500	423	465	491	502	391	502	498	480	460	436	444	453
	Sand Compression	7 days	1 513	1 928	2 000	2 801	2 522	2 863	2 962	3 382	2 106	2 254	2 852	3 198	1 837	2 126	1 858	2 481	1 982	1 942	2 796	2 886
		28 "	2 396	3 404	2 909	4 176	3 445	3 713	4 270	4 468	3 226	3 377	4 356	4 153	2 913	3 462	3 661	3 881	3 524	3 083	4 311	3 818
		90 "	2 844	4 835	4 896	4 684	4 388	3 872	4 692	4 702	3 799	4 947	4 786	5 420	2 508	4 134	4 311	5 571	4 226	3 605	5 663	4 698
		180 "	2 877	3 242	3 846	3 654	3 312	3 775	3 633	3 529	3 587	3 246	3 971	3 933	3 443	3 806	4 711	4 292	4 740	4 390	6 238	5 033
Damp Closet	Concrete	7 days	1 043	1 390	1 602	2 235	1 655	1 982	1 890	1 863	1 622	1 747	2 355	2 265	1 592	1 657	1 720	1 897	1 460	1 203	2 093	1 983
		28 "	1 762	2 360	2 438	3 282	2 675	2 738	2 402	2 380	2 418	2 608	2 858	3 093	2 285	2 762	3 152	3 015	2 593	2 483	3 620	2 803
		90 "	2 590	3 027	3 523	3 908	3 425	2 835	2 632	2 923	3 330	3 223	3 468	3 848	3 298	3 552	3 557	3 979	3 472	2 942	4 058	4 288
		180 "	2 112	3 100	3 017	3 962	3 007	3 182	2 878	2 972	3 417	3 577	4 065	4 297	3 390	3 640	4 248	4 347	4 413	3 327	4 038	4 663
Combined Storage	Neat Tension	28 days	540	565	....	302	405	365	303	253	596	469	341	286	481	507	285	465	419	533	423	400
		90 "	571	694	....	662	669	427	407	662	635	750	566	616	718	718	666	669	866	818	187	528
	Neat Compression	28 days	9 612	10 933	....	....	10 321	13 050	10 346	11 029	11 783	11 167	9 908	12 400	9 888	10 888	10 850	11 241	11 643	11 292	17 054	15 758
		90 "	12 592	12 638	....	....	13 233	14 541	11 108	15 229	13 788	12 604	13 454	15 592	....	12 892	13 983	13 029	15 279	14 888	17 667	16 188
	Sand Tension	28 days	467	403	452	517	513	600	624	637	483	444	485	572	391	520	491	552	482	486	545	542
		90 "	504	496	653	689	563	633	446	693	563	570	603	643	565	546	513	651	562	526	587	613
	Sand Compression	28 days	3 346	3 450	4 458	5 725	4 688	5 217	5 304	5 116	3 867	3 871	4 450	4 583	3 016	3 721	3 950	4 063	2 888	3 138	6 267	4 917
		90 "	4 279	4 554	5 454	7 008	5 629	6 162	6 038	7 438	4 358	5 488	5 646	6 379	4 312	4 504	5 171	6 425	4 042	4 204	6 533	6 354

TABLE III (*Continued*).—STRENGTHS DEVELOPED BY VARIOUS SPECIMENS.

ALL VALUES, WHICH ARE IN POUNDS PER SQUARE INCH, ARE THE AVERAGE OF THREE SPECIMENS.

Kind of Specimen.		Tested at end of	Cement No. F 1	F 2	F 3	F 4	Cement No. G 1	G 2	G 3	G 4	Cement No. H 1	H 2	H 3	H 4	Cement No. J 1	J 2	J 3	J 4	Cement No. K 1	K 2	K 3	K 4
Stored in Water	Neat Tension	24 hours	500	216	423	432	326	170	428	291	403	455	403	451	287	143	322	376	318	165	298	209
		7 days	672	692	714	729	772	663	807	793	709	859	810	798	647	596	532	845	542	394	557	610
		28 "	671	760	767	766	768	909	782	866	763	768	732	816	698	848	667	703	616	709	623	712
		90 "	721	879	687	843	880	918	833	927	857	723	672	770	751	859	719	853	708	787	737	650
		180 "	769	881	761	795	801	700	631	793	787	733	667	805	696	785	668	819	778	715	699	708
	Neat Compression	7 days	8 980	8 452	9 855	10 389	8 969	5 371	9 755	10 328	6695	7 864	7 798	8 208	6 997	7 933	8 124	10 162	6 543	5 619	6 571	8 871
		28 "	11 165	10 687	11 662	12 835	11 247	11 798	11 603	11 664	7697	9 527	8 046	9 150	10 891	11 900	11 038	13 782	8 788	9 198	8 427	11 302
		90 "	12 812	14 696	11 904	14 733	11 167	12 687	13 083	14 788	9712	10 833	10 779	11 171	11 256	12 133	12 292	15 433	10 046	12 771	10 088	11 638
		180 "	14 226	14 759	16 652	17 868	15 458	14 868	17 492	15 829	11 642	12 821	12 219	12 454	13 604	16 346	16 412	18 158	12 079	15 812	13 242	17 578
	Sand Tension	7 days	259	293	347	376	303	278	363	324	327	378	310	391	211	282	324	372	270	156	258	278
		28 "	429	445	464	472	411	389	466	479	373	440	423	476	293	330	381	348	331	352	435	445
		90 "	458	473	527	592	499	454	529	481	412	529	460	547	429	483	472	482	423	439	560	428
		180 "	478	455	497	490	438	451	503	481	461	498	504	506	470	451	523	575	402	442	510	486
	Sand Compression	7 days	2 579	2 000	2 668	2 602	2 614	1 887	3 222	3 258	1928	2 387	3 120	2 778	1 871	2 048	2 780	3 078	1 787	1 383	2 385	2 339
		28 "	3 835	3 088	3 800	3 799	4 227	3 038	4 473	4 831	3078	2 962	3 684	3 358	2 782	3 106	3 811	4 734	2 850	2 629	3 485	3 582
		90 "	5 015	4 850	5 375	5 267	4 350	4 329	4 671	5 663	3825	3 654	4 400	4 250	3 962	3 974	5 446	5 467	4 162	3 021	4 533	4 867
		180 "	4 887	4 733	5 657	4 919	5 129	4 090	6 375	4 947	4 167	4 404	5 996	5 350	4 892	4 696	5 646	6 279	3 992	4 738	5 446	6 848
Damp Closet	Concrete	7 days	1 723	1 658	1 940	1 785	1 462	1 805	2 380	2 585	1910	2 102	2 200	2 385	1 370	1 552	2 242	2 388	1 325	1 112	1 973	1 515
		28 "	2 748	2 715	3 257	3 143	2 372	2 748	3 555	3 700	2590	2 965	3 215	3 033	2 637	2 812	3 657	3 337	2 087	2 155	2 877	2 498
		90 "	3 788	3 530	4 275	4 035	3 347	3 272	4 640	4 240	3777	3 757	4 527	4 227	3 022	2 910	3 900	3 462	2 584	2 701	3 698	2 686
		180 "	4 133	3 778	4 293	4 173	3 653	3 732	4 623	4 902	4 098	3 857	4 383	4 567	3 835	3 870	4 990	4 997	2 837	3 420	4 478	3 628
Combined Storage	Neat Tension	28 days	329	360	375	283	444	485	282	383	407	337	225	400	687	575	847	740	486	513	392	471
		90 "	566	687	600	561	762	851	638	714	707	804	592	650	668	828	806	731	556	742	632	513
	Neat Compression	28 days	13 446	12 188	14 433	12 125	11 408	12 421	13 762	15 479	8354	9 925	10 788	9 992	9 233	12 112	12 192	11 467	9 000	9 158	10 858	13 075
		90 "	15 075	14 050	16 083	16 205	12 721	13 271	13 579	15 254	10 379	9 779	11 621	11 600	10 779	13 883	15 275	14 567	11 258	12 638	10 729	10 900
	Sand Tension	28 days	491	542	583	550	605	503	599	501	587	484	504	607	529	568	510	605	439	406	533	503
		90 "	566	554	664	687	537	574	640	602	490	604	613	749	427	503	507	552	453	416	558	634
	Sand Compression	28 days	4 275	4 371	5 717	6 058	4 358	4 571	5 988	6 846	4392	4 229	5 875	5 475	4 154	4 708	4 650	4 571	3 592	3 317	4 871	4 129
		90 "	4 663	4 746	6 271	6 588	5 542	4 842	6 767	7 475	4 846	4 917	5 788	6 750	4 462	4 479	5 413	6 188	4 588	4 562	5 550	4 483

of this extra fineness, they have been stored as long as the others, have had more water used in the mixing, were more impervious, consequently drying out more slowly, and should have developed a strength at least equal to the coarser-cement specimens. If, on the other hand, it is due to more rapid and complete drying out, then the coarser-cement specimens should show the greater strength, since it is quite apparent that the latter are the more pervious from a visual examination of a broken briquette. The sand specimens being more pervious have allowed more complete hydration during the storage in water and a more complete drying out during the storing in air than the neat specimens; moreover, the hydration has apparently proceeded further with the finer-ground samples and after drying out, they show greater strength than the coarser-ground ones.

Considered from the colloidal theory of hardening, such a combined hardening of the sand specimens is ideal. During the period of water storage the hydration has been carried to such an extent that any further storage in this manner would bring about but slow changes; this hydration being further colloidal results in a soft and not very adhesive mineral glue. But on storing in air this glue dries out to an adhesive dry mass, giving much greater strength than in the wet condition. It further has stopped the growth of crystallin matter,—which even the most ardent supporter of the colloidal theory acknowledges does take place in time,—and consequently has stopped the disruptive action which these crystals may presumably produce.

These statements are not to be construed as advocating the "combined storage" of specimens. In the present investigation, the specimens were made and stored at nearly the same time, nevertheless the neat tension test specimens showed greater variations than the specimens stored in water. Specimens stored according to these methods would undoubtedly show great differences in those periods of the year, or between those localities, where humidity varied considerably.

Concrete Specimens.—With one exception the concrete made from the finer-ground cements shows the better strengths; further the addition of plaster to the finer-ground cements has not deleteriously affected the strength of the latter, though in the case of the coarser-ground cements the opposite may be said to

be the case to a very slight extent. The concrete made of six of the cements shows the lowest strength at the end of 90 days, resulting from the addition of plaster to the coarser-ground material; whereas, four cements show the highest strengths resulting from both the use of the finer-ground cement plus plaster. The grinding alone has produced the highest strength with five cements. One cement has not been improved by any of the three treatments.[1]

TABLE IV.—RATING OF THE FOUR GROUPS OF CEMENTS.

		Number of Specimens showing the Highest Strength at the end of				Number of Specimens showing the Second Highest Strength at the end of				Number of Specimens showing next to Lowest Strength at the end of				Number of Specimens showing the Least Strength at the end of			
		24 hrs.	7 dys.	28 dys.	13 wks.	24 hrs.	7 dys.	28 dys.	13 wks.	24 hrs.	7 dys.	28 dys.	13 wks.	24 hrs.	7 dys.	28 dys.	13 wks.
As received	Neat tension	2	0	0	1	1	1	0	0	4	4	5	7	2	5	5	2
	Neat compression	..	0	0	1	..	0	0	1	..	4	4	3	..	6	6	5
	Sand tension	..	0	0	0	..	0	0	1	..	5	3	2	..	5	7	7
	Sand compression	..	0	1	0	..	0	0	0	..	4	4	6	..	6	5	4
	Concrete	..	0	0	1	..	0	1	0	..	3	2	6	..	7	7	3
Plastered	Neat tension	3	1	5	5	1	2	4	3	1	4	1	1	5	3	0	1
	Neat compression	..	0	3	2	..	3	4	2	..	4	2	4	..	3	1	2
	Sand tension	..	1	0	1	..	3	3	4	..	3	4	3	..	3	3	2
	Sand compression	..	0	0	0	..	1	0	2	..	5	5	2	..	4	5	6
	Concrete	..	1	1	0	..	0	0	1	..	6	7	3	..	3	2	6
Ground	Neat tension	2	3	0	2	3	5	1	3	2	0	4	0	2	2	5	5
	Neat compression	..	1	3	0	..	6	1	6	..	2	3	2	..	1	3	2
	Sand tension	..	1	2	3	..	6	5	2	..	2	3	5	..	1	0	0
	Sand compression	..	3	3	4	..	6	7	5	..	1	1	1	..	0	0	0
	Concrete	..	4	6	5	..	6	3	4	..	0	1	0	..	0	0	1
Ground-plastered	Neat tension	3	6	5	2	5	2	5	4	2	2	0	2	0	0	0	2
	Neat compression	..	9	4	7	..	1	5	1	..	0	1	1	..	0	0	1
	Sand tension	..	8	8	6	..	1	2	2	..	0	0	2	..	1	0	0
	Sand compression	..	7	6	6	..	3	3	3	..	0	0	1	..	0	0	0
	Concrete	..	5	4	4	..	4	6	5	..	1	0	1	..	0	1	0

The percentage of gain of strength of the various specimens for each cement, at the end of the 90-day period, is somewhat less for the finer-ground cements. In other words, the fine-ground cements are not gaining as much strength as the coarse-ground. It is too early in the investigation to say whether this will continue or not; it is frequently stated that in time, the strength of concrete from coarse-ground cement will

[1] The concrete of this cement has been examined to see if possibly there was an error in marking the specimens. By chemical analysis it was found that so far as the SO_3 content was concerned, the specimens were properly marked; whether they were marked properly in regard to fineness is yet in doubt, though it is intended to examine the mortar of these cements for relative size of unhydrated cement particles, to determine this point.

equal that of concrete made from the same cement ground finer, thus placing all the benefit to be obtained from fine grinding in the early periods.

In Table IV is given a summary which shows the number of specimens of each group of cement (as received, ground finer, SO_3 added, and ground finer and SO_3 added), which gave the highest, second highest, third highest and lowest strengths at the various periods. This brings out very clearly a number of the points made in the above discussion of the effect produced on the strength of the various specimens by the different further treatment of the original cements. Thus, under the heading "number of specimens showing the highest strength at end of 13 weeks," it is seen that but one neat tension, one neat compression, and one concrete specimen of the cements, as received, ranked higher than any of the other three samples prepared from the same brand; whereas, at the same period two neat tension, seven neat compression, six sand tension, six sand compression, and four concrete specimens of the cement which was finer ground and had more SO_3 added, ranked higher than any of the other samples of the same brand.

COEFFICIENT OF EXPANSION.

In Table V are shown the results of the measurements of neat and 1:3 standard sand mortar bars to determine the coefficient of expansion due to hydration. These bars were 13 in. long by 1 sq. in. in cross-section, and had a gage length of 10 in. The measurements were made with a Berry strain-gage and are not corrected for temperature variation, since there are no reliable figures for the coefficient of thermal expansion of neat cement or mortar; however, there has never been a temperature variation of more than 6° C.

The first measurement was made at the end of 24 hours, the use of a Berry strain-gage not permitting a reading before this period. The results, therefore, show the change in length which has taken place since the final set took place. Unless otherwise noted, the results are the average of several readings on duplicate bars.

From the results of the neat specimens, it is clearly seen that the cements, as received, show by far the least expansion; the

finer-ground cement the next least; and that the addition of the plaster has increased the expansion of the coarse cement to a much greater extent than the fine cement. If the neat bars were

TABLE V.—EXPANSION OF NEAT AND 1:3 MORTAR BARS.

Cement No.	Percentage of Expansion of Neat Specimens at the end of					Percentage of Expansion of Mortar Specimens at the end of				
	3 days.	5 days.	1 week.	4 weeks.	13 weeks.	3 days.	5 days.	1 week.	4 weeks.	13 weeks
A-1	0.012	0.019	0.016	0.046	0.072	−0.002	0.009	−0.002	0.008	0.019
A-2	0.028	0.036	0.036	0.078	0.103	−0.006	0.004	−0.004	0.004	0.012
A-3	0.014	0.020	0.014	0.051	0.080	−0.003	0.005	0.001	0.010	0.016
A-4	0.022	0.033	0.028	0.069	0.094	−0.004[a]	0.004	−0.002	0.007	0.012
B-1	0.010	0.017	0.021	0.051	0.074	0.003[a]	0.001	0.002	0.012	0.009
B-2	0.012[a]	0.027	0.029	0.059	0.082	−0.001	0.001	0.002	0.007	0.005
B-3	0.008	0.023	0.029	0.061	0.087	0.002	0.004	0.004	0.012	0.012
B-4	0.016	0.028	0.035	0.062	0.084	0.000[a]	0.004	0.002	0.012	0.011
C-1	0.010	0.015	0.016	0.042	0.057	−0.001	0.001	0.000	0.008	0.005
C-2	0.021	0.025	0.029	0.056	0.067	0.001	−0.001	0.001	0.008	0.005
C-3	0.009	0.015	0.019	0.041	0.056	0.002	0.000	−0.001	0.007	0.005
C-4	0.010	0.019	0.024	0.046	0.061	−0.002	−0.001	0.001	0.007	0.008
D-1	0.019	0.025	0.030	0.056	0.077	0.003	0.001	0.000	0.011	0.014
D-2	0.027	0.034	0.039	0.067	0.087	0.004	0.001	0.009	0.013	0.015
D-3	0.020	0.027	0.033	0.062	0.087	0.003[a]	0.001	0.003	0.015	0.016
D-4	0.029	0.037	0.043	0.075	0.101	0.006	0.002	0.005	0.015	0.014
E-1	0.009	0.017	0.018	0.035	0.057	−0.003	0.001	−0.004	0.008	0.008
E-2	0.047	0.068	0.072	0.121	0.142	−0.002	0.004	−0.003	0.009	0.009
E-3	0.015	0.024	0.025	0.057	0.080	0.002	0.006	0.001	0.012	0.011
E-4	0.040	0.052	0.055	0.089	0.111	−0.001	0.002	−0.002	0.010	0.011
F-1	0.013	0.020	0.026	0.048	0.063	−0.007	−0.002	0.001	0.008	0.005
F-2	0.057	0.070	0.076	0.106	0.124	0.010	0.014	0.015	0.023	0.021
F-3	0.019	0.026	0.026	0.053	0.073	0.000	0.003	−0.002	0.010	0.009
F-4	0.046	0.054	0.058	0.084	0.101	−0.006[a]	−0.002	−0.006	0.002	0.002
G-1	0.015	0.031	0.023	0.061	0.085	−0.004	0.005	−0.001	0.013	0.017
G-2	0.063	0.094	0.104	0.193	0.191	0.005	0.024	0.024	0.034	0.040
G-3	0.020	0.029	0.023	0.060	0.070	−0.003	0.008	0.002	0.015	0.020
G-4	0.064	0.088	0.091	0.157	0.196	−0.006[a]	0.010	0.007	0.015	0.019
H-1	0.028	0.034	0.039	0.065	0.088	0.006	0.004	0.011	0.015	0.015
H-2	0.031	0.038	0.042	0.069	0.090	0.002	0.001	0.001	0.009	0.010
H-3	0.020	0.032	0.029	0.069	0.094	−0.004[a]	0.008	−0.002	0.009	0.010
H-4	0.021	0.035	0.033	0.068	0.091	−0.004	0.007	0.002	0.011	0.015
J-1	0.018	0.028	0.031	0.052	0.068	0.001	0.005	0.001	0.012	0.009
J-2	0.069	0.084	0.085	0.097	0.117	0.008	0.009	0.005	0.015	0.012
J-3	0.024	0.035	0.035	0.057	0.087	0.005	0.005	0.001	0.008	0.008
J-4	0.029	0.037	0.035	0.063	0.079	−0.003	0.000	−0.005	0.005	0.005
K-1	0.041	0.052	0.061	0.095	0.120	0.009	0.009	0.011	0.021	0.024
K-2	0.178	0.317	0.393	0.509	0.535	0.090[a]	0.134	0.131	0.148	0.152
K-3	0.057	0.064	0.072	0.107	0.132	0.011	0.011	0.013	0.025	0.027
K-4	0.150	0.219	0.224	0.270	0.288	0.065	0.063	0.063	0.074	0.077

[a] Measurements made on one bar only.

alone considered there would be some reason for alarm for the cement containing the added SO_3. It possibly would be expected that cements, which in the neat show an expansion of 0.10 to 0.53

per cent, would certainly disintegrate, no matter whether they were used as a mortar or concrete. But an examination of the results from the mortar bars does not show anything very striking, other than possibly the fact that a cement which shows a very large expansion of the neat specimens may show a less expansion as a mortar than a second cement which gave a smaller expansion as a neat specimen. Thus we have samples A-1 and A-2 showing an expansion of 0.072 and 0.103 per cent as neat specimens, but as mortar specimens they show 0.019 and 0.012 per cent, respectively.

The very large coefficient of expansion of hydration of the cements containing more SO_3 might be used as an argument in their favor. Many other investigators have shown that specimens stored in air contract, and according to some results, do so to a remarkable degree.[1] As these high-SO_3 cements show a large expansion due to hydration in water, it is reasonable to suppose that when aged in air the contraction would be less than low-SO_3 cements, and consequently the very undesirable shrinkage, which is held to be the cause of hair cracks and stresses in the reinforcing, might be naturally reduced.

While the neat specimens show that the arrangement of the four groups of cements in the order of increasing expansion would be (1) coarse ground, (2) ground finer, (3) ground finer plus SO_3, and (4) coarse ground plus SO_3, the mortar specimens do not appear to follow any law. Cements with SO_3 added do show a tendency to greater expansion, but it is not very marked except with the white cement, and on the whole the mortar specimens do not show anything conclusive.

The rate of increase of the expansion with aging is falling off markedly, and would lead to the conclusion that no injurious effect may be expected, since now the process of hydration of those constituents which give the late strength is going on to such a degree, that their cohesive strength will be sufficient to counterbalance the disruptive effects which are shown by the increase in length. In other words, unless the expansion at the early

[1] McMillan, "Shrinkage Stresses and the Columns of the Edison Building," *Engineering News*, Vol. 73, No. 10 p. 502; White, "Destruction of Cement Mortars and Concrete through Expansion and Contraction," *Proceedings*, Am. Soc. Test. Mats., Vol. XI, p. 531 (1911), and "Volume Changes in Portland Cement and Concrete," *Proceedings*, Am. Soc. Test. Mats., Vol. XIV, Part II, p. 203 (1914).

period is much greater than that shown, there will be no injurious effects produced at the late periods. This however is a matter of conjecture and until more results on the measurement of the early expansion of cements which later disintegrate are available, nothing definite can be stated.

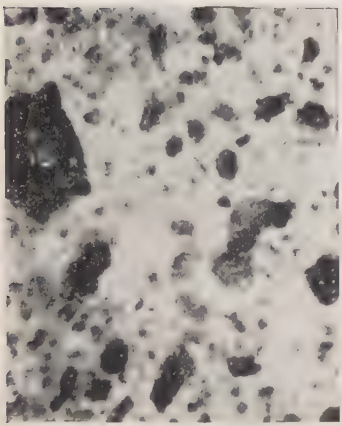
Effect of Composition and Constituents.

One reason for the use of the particular brands mentioned was the fact that they offered quite a range in composition, and it was thought that they might for this reason exhibit the effect of the treatments differently. Only to a very limited extent is this true. Thus apparently the lowest silica cement has been benefited more by all of the treatments, than any of the others. This, however, is also a high-alumina cement. From all of the results obtained with the white cement, which is the highest silica and lowest alumina cement, it is shown that fine grinding alone produced the most marked improvement; but in addition to the silica and alumina content, the other variables must be considered before any definite conclusion can be drawn. The very low amount of iron oxide in the white cement really puts the latter in a class by itself, and consequently the effects produced in it by the treatments cannot be compared with the effects produced in the other cements. Cements E and F, which have very similar composition, have been affected rather similarly. The presence of the high-iron-oxide and alumina content of J does not seem to have made it act differently from the other cements. Cements B and G, which are fairly similar in composition, give rather different results when treated. On the whole, therefore, the study of the composition in connection with the treatment does not lead to any definite conclusion.

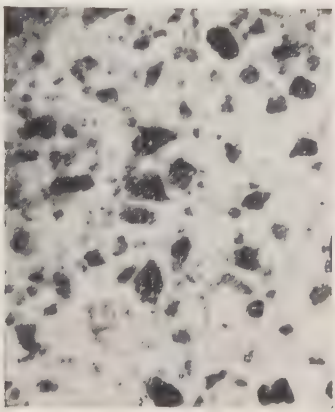
The unground clinker of none of the cements was available, consequently a study from the viewpoint of constituents could not be made in the most advantageous manner. The cements were all examined petrographically and it was found that one of them contained free lime; all contained some carbonate of lime in very small amounts. The only aluminate present was $3\ CaO.Al_2O_3$, except in cements A and J, where very small amounts of $5\ CaO.3\ Al_2O_3$ were noted. All contained $3\ CaO.SiO_2$, varying from a very small amount in cement K to rather large amounts

in B, E, F, G and H; but in every case the predominating constituent was β $2CaO.SiO_2$. It was not possible to make the study quantitative, neither was it possible to determine the physical appearance of the clinker—whether of a decided vitreous or of a soft coke-like nature. Consequently, it is as impossible to explain the action of the cement in hydrating from the standpoint of constituents as of composition.

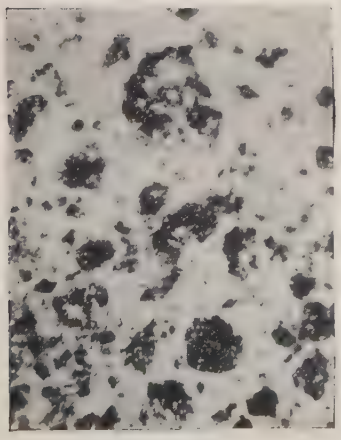
In Fig. 1 are shown four photomicrographs of polished sections of neat briquettes at the end of 90 days. It will be noted that two of these were made from cements as received, while the other two were made from the same cements ground fine. The difference in particle sizes is immediately noticeable. It is also noticed that there is no distinct outline of the dark colored particles, which with a few exceptions are the unhydrated constituents—these exceptions being voids in the briquettes. This lack of outline is due either to hydration of the edge or embedment of the particles in hydrated material. It can also be realized what difficulty and inaccuracy there would be in any attempt to determine quantitatively the percentage of this unhydrated material, either from such a photomicrograph or any projection of the enlarged surface of the thin section upon a ground glass. The latter was attempted in the case of a number of sections of 90-day briquettes. After placing the section on the stage of the microscope, the field enlarged to 135 diameters was examined on the ground glass of a camera. It was decided from the general conditions that the use of a planimeter, in a dark room directly on the ground glass, to integrate the areas was too unsatisfactory to attempt. The image was consequently thrown upon a sheet of tracing paper, fastened on a clear glass in place of the ground glass, and the various areas representing the hydrated and unhydrated material and the voids of the sections were outlined upon it, and attempts made to integrate them with a planimeter. It was found, however, that it did not give accurate enough results, particularly with the very small areas, even when the most advantageous arm length of the planimeter was used. Consequently, the final method of integration used was to weigh the paper representing the entire area on an analytical balance, and then cutting out by means of a sharp-pointed pin the various areas and weighing these. From this procedure it was possible



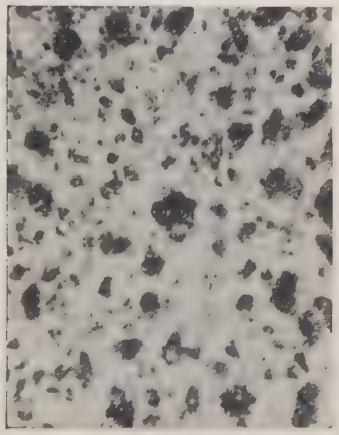
B-2.



B-4.



C-1.



C-3.

FIG. 1.—Photomicrographs of Polished Sections of Four Neat Briquettes Tested at the End of 90 Days. Magnification, × 80.

to calculate the percentages of each area, and consequently the percentages of unhydrated and hydrated constituents.

The results of a number of such integrations are given in Table VI. These results are undoubtedly slightly low for the unhydrated material, since it is assumed that all of the latter is brown or black in color, whereas it is only those constituents of the clinker which contain iron that are so colored; the tricalcium silicate and tricalcium aluminate are always white and would not be distinguished in sections examined by reflected

TABLE VI.—AMOUNT OF HYDRATED MATERIAL IN SOME 90-DAY NEAT BRIQUETTES.

Cement No.	Field No. 1 (Corner)			Field No. 2 (Center)			Field No. 3 (Between)			Average		
	Area of Voids, per cent.	Void-Free Area.		Area of Voids, per cent.	Void-Free Area.		Area of Voids, per cent.	Void-Free Area.		Area of Voids, per cent.	Void-Free Area.	
		Hydrated Material, per cent.	Unhydrated Material, per cent.		Hydrated Material, per cent.	Unhydrated Material, per cent.		Hydrated Material, per cent.	Unhydrated Material, per cent.		Hydrated Material, per cent.	Unhydrated Material, per cent.
H-1	3.8	70.0	30.0	6.4	69.1	30.8	5.6	70.1	29.9	5.3	69.7	30.2
H-2	6.2	72.2	27.8	3.4	65.3	34.6	9.1	69.7	30.2	6.2	69.1	30.9
H-3	3.9	76.1	24.0	5.2	76.6	23.5	1.5	76.0	24.0	3.5	76.2	23.8
H-4	7.2	79.8	20.2	5.2	73.1	26.9	3.4	74.9	25.2	5.3	75.9	24.1
E-1	4.8	69.8	30.2	6.7	66.6	33.3	8.2	67.1	33.0	6.6	67.8	32.2
E-2	9.2	64.2	35.8	5.7	61.4	38.6	10.1	64.6	35.4	8.3	63.4	36.6
E-3	8.5	70.9	29.2	9.8	67.2	32.8	5.5	69.0	31.0	7.9	69.0	31.0
E-4	10.7	67.0	33.0	10.9	67.9	32.2	8.2	69.6	30.4	9.9	68.2	31.9

light, from the hydrated particles. The latter two constituents are however the quick-hydrating particles of clinker, and the amounts of these unhydrated at the end of 90 days are small.[1] It should also be stated that the figures given represent the average of three fields of each section; one field was within $\frac{1}{8}$ in. of the edge of the briquette, a second field at about the center, and the third midway between the other two fields.

[1] Klein and Phillips, "Hydration of Portland Cement," *Technologic Paper No. 43*, Bureau of Standards.

From the two cements examined it appears that the fine grinding has caused a greater amount of hydration; in one case this is rather pronounced, but in the other case it is slight. If it were attempted to correlate these results with the strengths at the end of 90 days, a rather anomalous conclusion would follow, since cements H 3 and H 4, which show more hydrated material than H 1 and H 2, have both shown retrogression since the 28-day period; whereas, cements E 3 and E 4 which differ, but not very greatly, from E 1 and E 2, both show marked gains in strength. Apparently therefore too much hydration produces retrogression, but in no case should conclusions be drawn from few or isolated results.

GENERAL CONCLUSIONS.

1. So far as time of set is concerned, no disadvantageous results have been produced. The fine grinding produces a quicker set; the additional SO_3 produces a quicker initial but slower final set. Few of the changes however are such as to place the treated cement beyond specification limits.

2. Either fine grinding, the addition of SO_3, or fine grinding plus addition of SO_3, generally improves the strength of neat specimens. The neat specimens, however, do not give very consistent results, and not much value should be placed upon them. The indications are that at later periods, the results for all neat specimens, including those made from the coarse cements, will be very close together.

3. The strength of the standard sand mortars in tension shows the advantage of all of the three treatments of the original cements. In the compression test specimens the addition of SO_3 to the coarse cement has caused a falling off of the rate of gain of strength, which might be assumed to be caused by steadily increasing disruptive action. Whether this will appear later with the finer-ground cements, to which the addition was made, cannot be foretold from the present results. It may appear with the long-time specimens, and again it may be that the finer grinding has produced sufficient strength to entirely counterbalance any disruptive action due to too much SO_3.

4. The "combined-storage" method of aging the small specimens has given results which are of interest to American

manufacturers and users of cement, since but very few results of tests conducted in this manner on American cements are available. These results are of more interest in this one particular, than in giving a satisfactory conclusion in regard to the value of finer grinding or further addition of SO_3. However, the neat specimens broken in tension do not show that the cements are improved by either of the treatments used; and the results with these compression specimens are not very conclusive; the sand tension test specimens do not show that they have been as markedly improved by the treatments as do the similar specimens normally stored; the sand compression test specimens are more conclusive and do show the value of finer grinding with or without addition of SO_3.

5. At the end of 28 days the grand average strength of all the concrete specimens, made from the cements as received, was 2417 lb. per sq. in.; of the specimens made of the cement with the additional plaster 2635 lb. per sq. in.; of the specimens made from the finer-ground cement 3103 lb. per sq. in.; and of the specimens made from the finer-ground cement further plastered was 3028 lb. per sq. in.; the strength of the concrete or these specimens at the end of 90 days was 3263, 3175, 3828 and 3760 lb. per sq. in., respectively. From these figures it appears that at the end of 28 days, by the above-mentioned treatment, the original amounts have been improved about 10, 28 and 25 per cent, respectively; and at the end of 90 days about 0, 17 and 15 per cent, respectively. It would appear, therefore, from these grand averages that at later periods the original cements may give as good strength values in concrete as the treated cements. It might even be assumed that the further SO_3 in the coarse cements was not desirable from the standpoint of strength. It is, however, very evident that finer grinding is very advantageous for early strength, and that the addition of more SO_3 to such finer-ground cements to more properly control their set, is not disadvantageous at this period.

6. The results of expansion measurements on neat specimens stored in water show large increases in length for the specimens made from the cements containing more SO_3. In the sand specimens this is not so apparent. At present these specimens do not furnish much aid in studying the problem under investigation.

It may be, however, that these neat specimens are foretelling the ultimate results which will be noticed in the strength specimens.

7. The results are not conclusive as to the effect of the composition upon finer grinding or further SO_3. Neither can any satisfactory conclusion be drawn from the constitution of the cements.

8. What results have been obtained in the study of the relative amounts of hydration, produced by the treatments in the 13-week neat tension test specimens, are not sufficient to make any positive statement. The study of this phase of the subject is being continued with the 26-week concrete specimens.

Acknowledgments are due to Mr. E. R. Gates for superintending the making and testing of all specimens, and also to Mr. A. A. Klein for the microscopical studies.

DISCUSSION.

Mr. Mattimore. MR. H. S. MATTIMORE.—I should like to ask Mr. Bates if the concrete that shows the least amount of unhydrated material was mixed longer than usual.

Mr. Bates. MR. P. H. BATES.—No, that is all hand-mixed concrete.

Mr. Mattimore. MR. MATTIMORE.—How long was it mixed?

Mr. Bates. MR. BATES.—It was turned over about a dozen times dry and then a dozen times wet.

Mr. Spackman. MR. H. S. SPACKMAN.—This paper emphasizes the danger of drawing conclusions from a single set of tests, or even from a limited number. Disregarding, in the interests of brevity, the results obtained on the pastes and mortars, and considering only the strength developed at the 90-day period by the concretes made from the cement as received and after grinding without addition of plaster of Paris, we find that if only cement B had been experimented with, the test could be advanced as proof that increasing the fineness on the 200-mesh sieve caused decrease in strength; while, on the other hand, if cement G only had been used, the test would indicate that an increase in fineness on the 200-mesh sieve of 9 per cent resulted in an increase in strength of the concrete at the 90-day period of 35 per cent. Similarly, if the tests had been confined to cements A, C and D, we could argue from the results that an increase of approximately 10 per cent in fineness on the 200-mesh sieve resulted in an average increase in the concrete at the 90-day period of approximately 6 per cent. On the other hand, if the tests had been confined to cements G, H and J, it would be clearly indicative that an increase in fineness on the 200-mesh sieve of approximately 11 per cent resulted in an average increase of almost 30 per cent in the strength of the concrete at 90 days.

The activity of a cement is a function of three distinct factors: (1) the chemical constitution and composition; (2) the degree of burning; (3) the fineness of grinding. In view of the effect of the other variables, it would seem improper to insist on

a uniform fineness for all cements, regardless of the manner of grinding, the chemical composition and constitution and the degree of burning; and such a course might result in actual injury to the quality of the cement, as indicated by cement B, and unnecessary expense in manufacture, without proportionate gain, as indicated by cements A, B, C and D. Mr. Spackman

It is interesting to note that the strength requirement automatically governs the fineness of grinding, the cements which were least improved by finer grinding being among the coarser cements when received. The requirement of a high fineness on the 200-mesh sieve is wrong, (1) because the sieving test is not a true measure of the fineness of grinding, as it does not show the content of the very fine flour, and (2) because it neglects the two other variables, which are equally important in governing the activity of the cement, that is, chemical composition and constitution, and degree of burning.

These tests are also interesting, in that they show no definite relation between either the tension or compression tests on pastes and mortars, and the strength developed by concretes made from the cement.

Mr. H. J. Force.—I should like to ask Mr. Bates whether the cements used passed the autoclave test. I should also like to ask if he can indicate the fineness of the raw material from which these cements were manufactured. Mr. Force.

Mr. Bates.—The samples were bought on the open market; whether they passed the autoclave test or not I do not know, as I did not make that test. The fineness of the raw mix is not known. It is not likely that a manufacturer would know the fineness of the raw mix of some of his product, bought some time after its manufacture, in a city three or four hundred miles from his plant. Mr. Bates.

I may say that Mr. Spackman's statements are in line with what the paper shows. We have ten different cements here originally ground to ten different finenesses; and then re-ground to 10 different finenesses. If you take one or two of them, you can prove that the fine grinding has not assisted at all in the grading of the concrete, whereas you can take others and prove that it has produced good results. I was sure the paper would bring that out before I started the work. The tendency in

Mr. Bates. investigations of this kind has been to show one or the other of these results. Some investigators showed conclusively that fine grinding was exceptionally desirable, and others showed that fine grinding was not desirable. This investigation has shown that it depends upon a number of variables. There are so many variables in the manufacture of cement, that to say that fine grinding is going to produce desirable results in all cements is wrong.

WEIGHT-VOLUMETRIC PROPORTIONING OF CONCRETE AGGREGATES IN TESTING.

By J. A. Kitts.

SUMMARY

The process of making concrete may eventually advance to such a stage of exactness that the proportioning will be done by weighing the materials rather than by the comparatively crude method of measuring them by volume. Then, as now, we should consider the volumetric proportions, as aggregates vary in character, specific gravity, percentage of voids, etc., so that certain weight (or volumetric) proportions suitable for one rock and sand are not adaptable to another.

The utility of a knowledge of the specific gravity and percentage of voids of a material, in determining volumes of that material by weighing, has not been appreciated. It is the object of this paper to describe the method of weight-volumetric proportioning, to point out its simplicity and importance, and to describe also a new method of progressive proportioning in opposition to the 1:1, 1:2, 1:3, etc., weight proportioning for cement-sand mortar, and 1:2:4, 1:3:6, etc., for concrete.

The metric system of weights and measures will be assumed in the formulas cited.

WEIGHT-VOLUMETRIC PROPORTIONING OF CONCRETE AGGREGATES IN TESTING.

By J. A. Kitts.

Error in Volumetric Proportioning.

Various methods have been devised for measuring volumes of cement, sand, and rock. One difficulty encountered in making a purely volumetric measurement of these materials is to place them in the vessel in such a manner that a uniform degree of compactness will obtain in each measurement. Another difficulty is due to the irregularity of the surface of the materials. In measuring a given volume the accuracy depends, necessarily, upon a single measurement. We may, however, determine the volume of a certain mass of material accurately by making several measurements.

If several determinations are made of the specific gravity and percentage of voids, and means are taken to obtain the full range of compactness in determining the percentage of voids, we may obtain a reliable figure for the apparent specific gravity of the material for average compactness. We may then weigh out volumes of the material with uniformity, and will have before us both weight and volumetric proportions, the volumetric proportions being the essential information.

Variability of Volume in Weight Proportioning.

From elementary principles we have:

Volume = Weight ÷ Specific Gravity.

We are not, however, dealing with actual volumes, in the case of cement, rock, and sand, but with apparent volumes:

Apparent Volume = Weight ÷ Apparent Specific Gravity, and
Apparent Specific Gravity = (Unit Volume − Voids) Specific Gravity, or
Apparent Volume = Weight ÷ (Unit Volume − Voids) Specific Gravity.

It is evident, therefore, that the apparent volume per unit of weight decreases as the specific gravity increases, and that it increases as the voids increase.

Table I shows the variations of the volumetric proportions of various sands in sand-cement mortars of normal consistency and of 1:3 proportions by weight; also the ratio of volume of cement paste to volume of voids in each case.

TABLE I.—VARIATIONS OF VOLUMETRIC PROPORTIONS IN 1:3 WEIGHT PROPORTIONING.

Sand No.	Specific Gravity.	Voids, per cent.	Apparent Specific Gravity.	Weight Proportions.	Volumetric Proportions.	Ratio of Cement Paste to Voids.
				Cement : Sand	Cement : Sand	
1[a]	2.65	37.0	1.67	1 : 3	1 : 3.18	0.85
2	2.74	35.2	1.78	1 : 3	1 : 2.97	0.95
3	2.63	39.8	1.58	1 : 3	1 : 3.34	0.75
4	2.78	36.6	1.76	1 : 3	1 : 3	0.91
5	2.77	27.9	2.00	1 : 3	1 : 2.64	1.36

[a] Standard Ottawa Sand.

Assuming that 110 lb. of cement make 1 cu. ft. of cement paste of normal consistency, the apparent specific gravity of cement is 1.76; and the volumetric proportions as indicated in Table I, are determined as follows:

$$\frac{\text{App. Volume Sand}}{\text{App. Volume Cement}} = \frac{3 \times \text{App. Sp. Gr. Cement}}{\text{App. Sp. Gr. Sand}} = \frac{5.28}{\text{App. Sp. Gr. Sand}}.$$

Is there any justification for comparing these sands in mixtures in which the proportions of voids filled with cement vary from 75 to 136 per cent? If it is desired to determine the relative strengths of sands per unit measure of cement, the volumes of sands should be equal. We are not so much concerned, however, with the strength of mixtures of equal volumetric proportions as we are with the comparative costs per unit of strength of the most economical mixtures with the various sands. In general, the minimum cost per unit of strength will obtain when the volume of cement paste is equal to that of the voids. The weights

and volumetric proportions of these sands for the ratio, Volume of Cement Paste ÷ Volume of Voids = Unity, will be indicated hereafter under "Comparative Tests of Sands."

WEIGHT-VOLUMETRIC PROPORTIONING.

If we wish to weigh out certain volumes of sand and cement for cement-sand mortar, with the object of using a certain volume of cement paste with a unit volume of sand, then:

$$\frac{\text{Weight of Cement}}{\text{Unit Weight of Sand}} = \frac{\text{App. Sp. Gr. Cement} \times \text{Proportion of Paste}}{\text{App. Sp. Gr. Sand}}.$$

Example: To use 0.37 parts by volume of cement paste of normal consistency with one part by volume of standard Ottawa sand, the part of cement by weight is $(1.76 \div 1.67)\ 0.37 = 0.390$. The weight proportions then are 1 sand: 0.39 cement, corresponding to volumetric proportions of 1 sand: 0.37 cement, or 1 cement: 2.7 sand.

The writer finds it desirable to use sand as the unit of measure, when considering a mortar, as in any stated volumetric proportions the ratio of volume of cement paste to volume of voids in the sand may be estimated at a glance. It will be observed in the above example that the volume of paste is equal to the proportion of voids in the sand.

Weight-volumetric proportioning of concrete aggregates may be made in a similar manner as will be indicated below under "Progressive Proportioning of Concrete Aggregates."

COMPARATIVE TESTS OF SANDS.

In making comparative tests of sands we should have like conditions in every case. If we have the same proportions by weight, or by volume, the ratio of volume of cement paste to volume of voids is different in every case. One mixture may be porous from too little cement while another may be dense but may have the sand particles widely separated by too much cement.

For like conditions, the consistency of the mixtures should be the same and the volume of cement paste should be equal to the volume of voids. The weight and volumetric proportions of sands Nos. 1 to 5 for comparative tests should be as shown in Table II for mortars of normal consistency.

It may be considered advisable to use an excess of paste in each case, for example, 105 per cent of the voids in the sands. The equation would then be:

$$\frac{\text{Weight of Cement}}{\text{Unit Weight of Sand}} = \frac{1.05 \text{ App. Sp. Gr. Cement} \times \text{Voids in Sand}}{\text{App. Sp. Gr. Sand}}.$$

The strength, or the cost per unit of strength, will indicate the most satisfactory sand.

TABLE II.—VOLUME OF CEMENT PASTE = VOLUME OF VOIDS.

Sand No.	Specific Gravity.	Voids, per cent.	Apparent Specific Gravity.	Volumetric Proportions. Sand : Cement	Weight Proportions. Sand : Cement	Ratio of Cement Paste to Voids.
1	2.65	37.0	1.67	1 : 0.370	1 : 0.390	1
2	2.74	35.2	1.78	1 : 0.352	1 : 0.348	1
3	2.63	39.8	1.58	1 : 0.398	1 : 0.443	1
4	2.78	36.6	1.76	1 : ..366	1 : 0.366	1
5	2.77	27.9	2.00	1 : 0.279	1 : 0.246	1

$$\frac{\text{Weight of Cement}}{\text{Unit Weight of Sand}} = \frac{\text{App. Sp. Gr. Cement} \times \text{Proportion of Paste}}{\text{App. Sp. Gr. Sand}}.$$

PROGRESSIVE PROPORTIONING OF SAND-CEMENT MORTAR.

In making a study of a given sand it is advisable that the mortar be made of the same consistency as that to be used in the field. The most satisfactory consistency for general use is the initial flowing consistency. Approximately 94 lb. of cement makes one cubic foot of cement paste of this consistency and the apparent specific gravity for this consistency is 1.5. This is the initial degree of wetness that will cause the cement paste to flow, as a liquid, from a plane inclined at 45 deg. The amount of water necessary to make a paste of this consistency is approximately as follows:

34 per cent of water by weight of dry cement.
39 " " " " volume " " "
50 " " " " " " resulting paste.

With this high percentage of water, necessary in the field, it will be recognized that the strength of a mortar of normal consistency is not representative of that of a mortar of the consistency under working conditions.

In order to determine the mortar of maximum density and also the mortar giving satisfactory strength, the progressive proportions in Table III are suggested, as preferable to the usual proportions, for sand No. 1 in a mortar of initial flowing consistency.

TABLE III.—PROGRESSIVE PROPORTIONS FOR CONSISTENCY UNDER WORKING CONDITIONS.

Proportions by Volume.		Ratio of Cement Paste to Voids.	Proportions by Weight.[1]
Cement Paste, per cent.	Sand : Cement Paste		Sand : Cement.
20	1 : 0.20	0.54	1 : 0.18
25	1 : 0.25	0.67	1 : 0.225
33.3	1 : 0.333	0.90	1 : 0.30
37[a]	1 : 0.37	1.00	1 : 0.333
40	1 : 0.40	1.08	1 : 0.36
50	1 : 0.50	1.35	1 : 0.45
66.7	1 : 0.667	1.80	1 : 0.60
100	1 : 1.00	2.71	1 : 0.90

a Volume of cement paste equal to volume of voids in sand.

[1] $\frac{\text{Weight of Cement}}{\text{Unit Weight of Sand}} = \frac{1.5 \times \text{Volume of Paste}}{1.67} = 0.9 \times \text{Volume of Paste.}$

The mortar of maximum density may be determined by weighing the test specimens.

PROGRESSIVE PROPORTIONING OF CONCRETE AGGREGATES.

Having determined the mortar of maximum density and satisfactory strength, progressive proportions of the mortar with the rock may be made in a similar manner as cement with sand.

An amount of rock equal to the volume of the test cylinder should be used for each test specimen.

The progressive proportions in Table IV are suggested for the following cement, sand and stone:

Material.	Specific Gravity.	Per cent of Voids.	Apparent Specific Gravity.
Cement	3.1	51	1.5
Sand No. 1	2.65	37	1.67
Rock	2.50	43.65	1.41

Test Cylinder: Diameter, 8 in.; length, 16 in.; volume, 13.1793 liters
Weight of Rock to fill Cylinder: $1.41 \times 13.1793 = 18.583$ kg.

TABLE IV.—PROGRESSIVE PROPORTIONS OF MORTAR WITH ROCK.

Weight of Rock, kg.	Ratio of Mortar to Voids.	Mortar of "Work" Consistency.				Volumetric Proportions.
		Per cent.	Volume, liters.	Weight of Sand,[1] kg.	Weight of Cement,[2] kg.	
18.583	0.80	35	4.673	7.804	2.593	1 : 2.7 : 7.7
"	0.92	40	5.272	8.871	2.926	1 : 2.7 : 6.8
"	1.00[a]	43.65	5.753	9.575	3.193	1 : 2.7 : 6.2
"	1.05	45	5.930	9.937	3.291	1 : 2.7 : 6.0
"	1.15	50	6.589	11.004	3.657	1 : 2.7 : 5.4

[a] Volume of mortar equal to volume of voids in rock.

[1] Weight of Sand $= \frac{\text{Volume of Mortar} \times \text{App. Sp. Gr. of Sand}}{\text{Yield of Mortar}}$

[2] Weight of Cement $= \frac{\text{Volume of Mortar} \times \text{Voids in Sand} \times \text{App. Sp. Gr. of Cement}}{\text{Yield of Mortar}}$.

The yield of mortar for sand No. 1 and 37-per-cent cement paste, as used in Table IV, is assumed as unity.

The weight of the test specimens will indicate the mixture of maximum density.

Permeability specimens may be made in the same proportions.

CONCLUSIONS.

By this method of weight-volumetric proportioning we may obtain a fairly accurate knowledge of the action of various combinations of materials in mortars or concretes and the results, instead of being freaky, will be consistent to a degree not attain-

able by simple volumetric or simple weight proportioning. The advantages of this method are:

1. That volumes may be weighed with ease and accuracy;
2. That the proportions may be found with regard to the superficial volume, actual volume, or the volume of voids of the materials;
3. That the error of volumetric measurement due to variability of compactness and irregularity of surface is eliminated;
4. That the voids in sand may be accurately filled with cement paste and the voids in rock with cement-sand mortar.

A porous mortar or a porous concrete is not an economical mixture with reference to cost per unit of strength. A mortar with cement greatly in excess of the volume of voids in the sand is not an economical mixture. A concrete with mortar greatly in excess of the volume of voids in the rock is not an economical mixture. The vital changes in density, permeability, and cost per unit of strength take place by variations in the volume of the filling medium when this volume is in the vicinity of that of the voids. In progressive proportioning of mortars or concretes we may investigate a wide range of proportions including that in which the voids in the materials are filled; or, we may investigate a limited range in the vicinity of the voids, by means of the methods proposed, and thus determine the most economical proportions accurately and scientifically.

DISCUSSION

Mr. Cloyd M. Chapman *(by letter)*.—Before entering upon a discussion of some of the statements in the paper which may be worthy of further consideration, I want to express hearty sympathy with the main idea of the author, which seems to be to encourage the more scientific and accurate proportioning of concrete based on the quality and grading of the sand and stone to be used in any particular case, rather than upon any so-called "standard" proportions such as 1:2:4 or $1:2\frac{1}{2}:5$, etc. I want also to fully endorse the idea, that in order to determine what proportions will give the best results, it is necessary to make tests on various proportions covering a limited range. Mr. Chapman.

These two main points seem almost beyond debate, but there are several minor points in the author's argument and conclusions which seem open to discussion. For instance, the statement which occurs in several forms that "the volume of a certain mass of material may be accurately determined," is liable to mislead one who has not had considerable experience in determining weights per unit volume of materials of this character. A certain sand placed in a measure by a particular method and by a given operator may weigh say 89 lb. per cu. ft., equivalent to 46 per cent voids for a sand whose specific gravity is 2.65. The same sand, placed in the same measure, by another method, by the same operator, may weigh 104 lb. per cu. ft., equivalent to 37 per cent voids, and each of these determinations may be repeated a dozen times by the same operator and show equal or greater variation. Who shall say what the volume of a certain mass of that sand is?

What is the volume of a hundred pounds of cement? Is it 0.9 cu. ft. or is it 1.2 cu. ft.? One result is as easily obtainable as the other, depending upon how much the cement is compacted in the container. Shall it be compacted as much as possible, or as little as possible, or shall we find the two extremes and take an average? The apparent volume, apparent specific

Mr. Chapman. gravity, and percentage of voids in a material like sand may vary as much as 50 per cent depending upon the degree to which it is settled down in the container. It is misleading, therefore, to state that a sand has a particular apparent volume, or apparent specific gravity or percentage of voids, without making it clear how the determinations were made. Clean quartz sands of varying degrees of fineness and moisture content will, in the hands of various operators using various methods of determinations, give weights per cubic foot all the way from 55 to 110 lb.—a variation of 100 per cent.

So it may have been wisdom instead of ignorance which caused the mixer foreman to interrupt with the question: "Wad-do-you-mean, per cent of voids?"

Another point which seems worthy of discussion is the apparent assumption that uniformity may be depended upon in raw materials and in the mixed concrete to within 5 per cent, as is indicated in the third paragraph of the paper under "Comparative Tests of Sands."

Even assuming that it is possible to determine in the laboratory, within 5 per cent, the proper proportions in which a particular concrete should be mixed, there is no assurance, usually, of the following conditions which vitally affect in a practical way the theoretical accuracy at which the author aims:

First, there is usually small means of knowing that the sand sample tested represents the lot from which it was taken within such limits as would not affect the amount of cement required more than 5 per cent.

Second, the variation from the sample in future shipments of sand to the job is often such that a 5 per cent margin is entirely inadequate.

Third, the making of concrete under present-day methods is not a process which insures uniformity of mixing or prevention of segregation while transporting and placing to a degree represented by 5 per cent.

Another matter worthy of all the emphasis that can be placed upon it is calling attention, as the author does in the first sentence of the Summary of his paper, to the crude method now in use for measuring concrete materials. No matter how carefully and accurately the desired proportions are determined,

by the author's method or any other method, there is no means of knowing under present-day methods whether the specified proportions are approximated within 20 per cent. This is a condition which demands a remedy and perhaps it will come to pass, as the author states, that "The process of making concrete may eventually advance to such a stage of exactness that the proportioning will be done by weighing the materials rather than by the comparatively crude method of measuring them by volume." Mr. Chapman.

MR. A. C. GOW (*by letter*).—The practical value of any laboratory investigation to the field man depends very largely on the questionable assumption that the material delivered on the job, day by day, is of the same grade and physical characteristics as the specimens tested. Shortly after Mr. Kitts left the Isthmus, sand troubles began, and daily inspection and analysis of all sands delivered at the Pacific Terminals was authorized. Mr. Gow.

The work consisted of a sieve analysis for the granulometric proportioning of the particles, a water displacement test for the percentage of voids, checked by the weight-specific-gravity method, and a test to determine the percentage of silt deposit, using the small X-section test-tube columnar method. From a portion of the same sample that was analyzed, three briquettes were molded by the weight-volumetric method of proportioning and tested to destruction for tension at the age of 7 days. In molding the briquettes, the voids were just filled with cement paste and each batch of sand mortar was mixed to a normal consistency. Hence the briquettes were uniform in one respect, namely, each sand mortar was at its maximum density.

Three elements entered the situation in affecting the strength of the briquettes: (1) the percentage of voids and therefore the percentage of cement paste to the percentage or body of sand; (2) the percentage of silt or fine particles passing a No. 100 screen; and (3) the relative proportion of large to small particles—the former tending to increase, the latter to decrease tensile strength.

Two charts in which the unit tensile strength (sand mortar one week old) was plotted against the percentage of voids (Fig. 1) and the percentage of silt deposit (Fig. 2), suggest

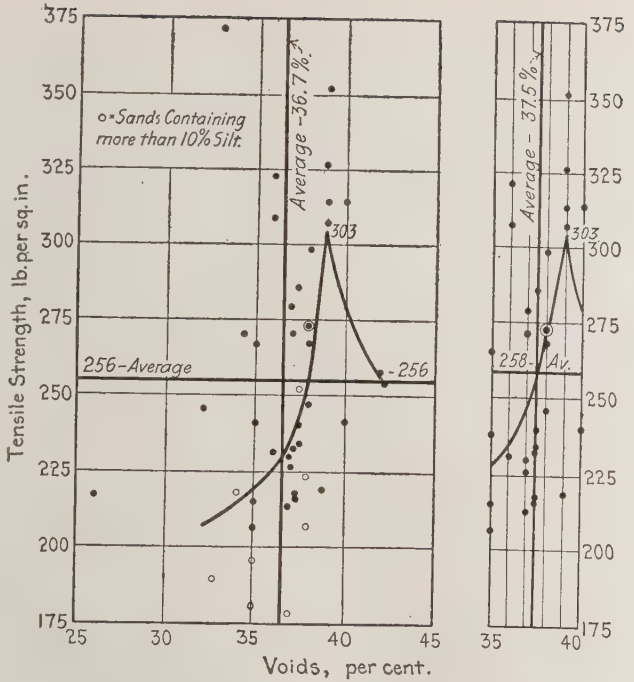


FIG. 1.—82 per cent of Tests Fall between 35 and 40 per cent of Voids. The Small Chart shows Tests Falling between These Limits and Omitting Tests made from Sand containing more than 10 per cent of Silt.

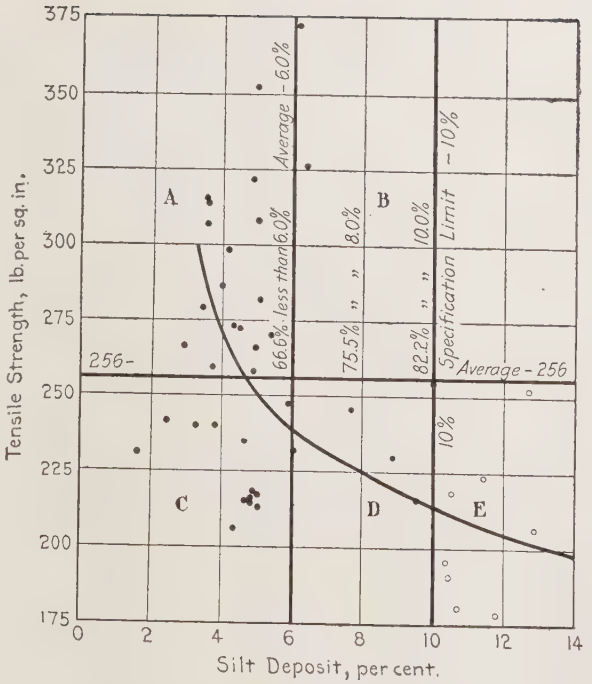


FIG. 2.—Curve showing Relation between Tensile Strength and Percentage of Silt Deposit.

several interesting tendencies. The curve of averages in Fig. 1 and the grouping of the tests in areas or zones of safety and danger in Fig. 2, deserve special attention. Mr. Gow.

The steady increase in the curve of averages till 39 per cent is reached, and the comparatively rapid fall afterwards, clearly defines the point of greatest efficiency, suggesting that for the sand in question, mortars of a mixture richer than $1 : 1\frac{1}{2}$ are not as efficient as maximum-density sand mortars of leaner mix.

In reviewing Fig. 2, areas *A* and *C* are safe, *D* is allowable, and *E* is condemned. The two tests in area *B* fall so close to the average line that practically they may be considered as belonging to area *A*. The evil effect of silt deposit when much greater than 6 per cent is clearly demonstrated. Effects of the granular make-up of the sand were reflected in individual specimens which appeared to contradict general tendencies rather than to show any influence on the series as a whole.

I describe this series of tests in detail to show the practical value of weight-volumetric proportioning of concrete for laboratory work. The mixing of maximum-density sand mortar for purposes of comparison seems at first to add another variable to the many uncertainties, but in reality I believe the reverse is true. Each mortar is at its maximum efficiency, hence, any variation of strength can be assigned definitely to one or a combination of the three elements known to affect its strength. The results of this series seem further to bear out this assumption.

MR. J. A. KITTS (*Author's closure by letter*).—It was not the object of this paper to cover the entire process of concrete proportioning and testing, as seems to be the impression of Mr. Chapman. Mr. Kitts.

Although the writer has made some extensive tests using this method, the results of which are indicated in the conclusions in this paper, the paper is in no way a record of tests. In the examples of proportions, materials were assumed with certain specific gravities and percentages of voids, and the proportions were obtained by mathematical determination, using the equations given.

The usual procedure may be followed in the determination

Mr. Kitts. of specific gravity and percentage of voids, and in the manner of treatment of the materials, drying, etc.

The writer has had no such difficulties in determining the specific gravity and percentage of voids, or the volume per unit of weight, as Mr. Chapman describes. Any of these determinations, or measurements, may be made with any desired accuracy depending upon the number of trials. Average compactness should be the condition sought, as stated in the paper, and no special measures need be taken for compactness if the materials are dry as they should be.

From the point of view of a concrete foreman I would say that Mr. Chapman's mixer foreman was ignorant.

No suggestion, or recommendation, was made in regard to an application of this method in the field. The 5-per-cent margin Mr. Chapman refers to was apparently taken from the 1.05 in the seventh equation. In progressive proportioning this may be varied, for example, 0.85, 0.90, 0.95, 1.0, 1.05, 1.10, 1.15, etc. This indicates the pliability of the method.

Mr. Chapman raises the usual question of the value of such tests in their applicability in the field, the variability of the raw materials, and other apparent but not real obstacles, making accurate and correct proportioning an impossibility in the field. The materials should, undoubtedly, be checked in the field for specific gravity and voids, and the laboratory tests would indicate the measures to be taken to correct the proportions in conformity with the changes in the materials. If the field tests indicated materials positively different from those tested, new tests should be made. The stock pile should be sampled far enough in advance so that no delays would be necessary.

The writer has prepared an article on "Weight-Volumetric Proportioning of Concrete Aggregates in the Field" to appear in *Western Engineering*, Vol. 6, No. 4, October, 1915. The following is, in part, an abstract of that article:

"*Field Determination of Specific Gravity and Proportion of Voids.*—Apparatus: Scales, water bucket, sheet of canvas. Method: (1) Weigh bucket, (*a*). (2) Weigh, individually, 5 or 10 buckets of material sampled from the storage pile, (*b*). Add a few buckets of material for wastage and thoroughly sun-dry. (3) Fill bucket with water and weigh, (*c*). Dry bucket. (4) Fill

bucket with dried material, carefully screeding same off level with brim, and weigh, (*d*). Pour material out on canvas sheet. (5) Fill bucket with an amount of water about equal to, or less than, the proportion of voids in the materials and pour in the material from the canvas, leveling same off without wastage. Add water to bring same to brim of bucket, weigh, (*e*). (6) Tabulate weighings as follows: Mr. Kitts.

Job..... Material..... Date..... By.....

No. of Test.	(*a*) Weight of Bucket.	(*b*) Weight of Bucket and Moist Material.	(*c*) Weight of Bucket and Water.	(*d*) Weight of Bucket and Dry Material.	(*e*) Weight of Bucket, Material and Water.	(*f*) Voids $\frac{e-d}{(c-a)}$	(*g*) Apparent Specific Gravity $\frac{d-a}{(c-a)}$	(*h*) Specific Gravity $\frac{g}{1-f}$	(*i*) Proportion of Water in Moist Material $\frac{b-d}{(c-a)}$	(*j*) Apparent Specific Gravity of Moist Material $\frac{b-a}{(c-a)}$
1.....										
2.....										
3.....										
4.....										
5.....										

Depending upon the showing of the laboratory tests, the proportions may be computed by use of the following equations:

$$\frac{\text{"Proportion of Sand}}{\text{Unit Proportion of Cement}} = \frac{1}{\text{Proportion of Voids in Sand}} \quad \ldots\ldots\ldots\ldots (8)$$

$$\text{Proportion of Rock} = \frac{\text{Proportion of Sand (8)}}{\text{Proportion of Voids in Rock}} \quad \ldots\ldots\ldots\ldots\ldots\ldots (9)''$$

The above equations would give proportions for maximum density. In case the laboratory tests show it necessary to use a proportion of cement in excess of the voids in the sand, in order to secure satisfactory strength, the yield of cement-sand mortar should be taken into consideration:

$$\text{"Yield of Mortar} = 1 + \frac{\text{Proportion Cement} - (\text{Proportion of Sand} \times \text{Voids})}{\text{Proportion of Sand}} \quad (10)$$

Equation (9) then becomes:

$$\text{Proportion of Rock} = \frac{\text{Proportion of Sand} \times \text{Yield of Mortar}}{\text{Proportion of Voids in Rock}} \quad \ldots\ldots\ldots (11)''$$

Mr. Kitts.

Equations (8), (9) and (11) will give a yield of concrete approximately equal to unity. The volumes for a mixer batch may be determined as follows:

$$\text{"Volume of Rock} = \frac{\text{Capacity of Mixer}}{\text{Yield of Concrete}} \quad \ldots\ldots (12)$$

$$\text{Volume of Sand} = \frac{\text{Capacity of Mixer} \times \text{Proportion of Sand}}{\text{Proportion of Rock} \times \text{Yield of Concrete}} \quad \ldots\ldots (13)$$

$$\text{Volume of Cement} = \frac{\text{Capacity of Mixer} \times \text{Proportion of Cement}}{\text{Proportion of Rock} \times \text{Yield of Concrete}} \quad \ldots\ldots (14)\text{"}$$

TABLE I.—PROGRESSIVE PROPORTIONS USED FOR CHAMÉ SAND.

Test No.	Parts by Volume. Dry Sand : Normal Cement.	Parts by Weight.[1] Dry Sand : Dry Cement.	Ratio of Cement Paste to Voids.
4a25	1 : 0.25	1 : 0.268	0.69
4a30	1 : 0.30	1 : 0.329	0.82
4a35	1 : 0.35	1 : 0.384	0.96
4a36.47[a]	1 : 0.3647	1 : 0.400	1.00
4a40	1 : 0.40	1 : 0.439	1.10
4a45	1 : 0.45	1 : 0.494	1.24

[a] Percentage of voids in sand.

[1] $\frac{\text{Weight of Cement}}{\text{Unit Weight of Sand}} = \frac{1.76 \times \text{Volumetric Proportion of Paste}}{1.604}$.

TABLE II.—AVERAGE RESULTS OF CHAMÉ CEMENT-SAND MORTAR TESTS.

Test No.	Tensile Strength, lb. per sq. in.		Weight, gr.		Number of Specimens.	
	7 day.	28 day.	7 day.	28 day.	7 day.	28 day.
4a25	159	221	131.09	131.98	4	4
4a30	218	242	132.23	131.71	4	4
4a35	243	294	135.20	135.06	4	4
4a36.47[a]	276	369	139.35	140.78	4	4
4a40	255	312	137.04	138.51	4	4
4a45	230	329	136.97	137.80	4	4

[a] Cement paste equal to voids, and mixture of maximum density.

The difficulty of using the exact quantities, or even approximating them, may be removed by weighing the materials in some manner, such as an occasional check on the barrows or carts handling the materials to the mixer:

"Weight of Volume required per Barrow = Volume required × Apparent Specific Gravity of Moist Material × 62.42..... (15)"

A Test in Weight-Volumetric Proportions.—The following sand test for the Division of Terminal Construction, Panama Canal, is given to support the conclusions in the paper; and to indicate the uniformity of the results of weight-volumetric proportioning: Mr. Kitts.

Chamê sand was tested for mortar of maximum density by using progressive proportions of cement with the sand and weighing the tension briquettes. The briquettes were then used in tension tests.

TABLE III.—COMPARISON OF AUTHOR'S THEORETICAL PROPORTIONS FOR MINIMUM COST PER UNIT OF STRENGTH WITH RESULTS OF PROF. WITHEY'S TESTS.

Material No.	Specific Gravity.	Voids, per cent.	Apparent Specific Gravity.	Theoretical Proportions for Minimum Cost per Unit of Strength.		
				Author's Computation.[1]	Withey's Tests.	
Sd. 1	2.66	36.5	1.69	1 : 2.63	Between 1 : 2 and 1 : 3, 1 : 2 minimum	
Sd. 2	2.74	35.2	1.78	1 : 2.88	" 1 : 2 " 1 : 3, 1 : 2 "	
Sd. 3	2.68	38.2	1.65	1 : 2.46	" 1 : 2 " 1 : 3, 1 : 3 "	(a)
Sd. 4	2.63	39.8	1.58	1 : 2.26	" 1 : 2 " 1 : 3, 1 : 2 "	(a)
Sd. 5	2.67	45.3	1.46	1 : 1.83	" 1 : 2 " 1 : 3, 1 : 2 "	(a)
Sd. 7	2.78	36.6	1.76	1 : 2.73	" 1 : 2 " 1 : 3, 1 : 2 "	
Sd. 8	2.70	36.4	1.72	1 : 2.59	" 1 : 2 " 1 : 3, 1 : 2 "	(a)
Sd. 9	2.75	36.0	1.76	1 : 2.78	" 1 : 2 " 1 : 3,	(a)
Sd. 10	2.77	27.9	1.99	1 : 4.07	" 1 : 3 " 1 : 4, 1 : 4 "	(a)
Sd. 11	2.72	35.0	1.77	1 : 2.87	" 1 : 2 " 1 : 3, 1 : 2 "	
Sg. 1	2.67	42.0	1.55	1 : 2.10	" 1 : 2 " 1 : 3, 1 : 2 "	(a)
Sg. 3	2.82	39.0	1.67	1 : 2.44	" 1 : 2 " 1 : 3, 1 : 2 "	(a)
Sg. 4	2.75	40.0	1.65	1 : 2.35	" 1 : 2 " 1 : 3, 1 : 2 "	(a)
Tg. 1	2.81	43.6	1.58	1 : 2.09	" 1 : 2 " 1 : 3, 1 : 3 "	

(a) Agreement with chart.

[1] $\frac{\text{Weight of Sand}}{\text{Unit Weight of Cement}} = \frac{\text{App. Sp. Gr. Sand}}{\text{App. Sp. Gr. Cement } (=1.76) \times \text{Voids in Sand}}$.

The percentage of voids in the sand was 36.47, the specific gravity 2.525, and the apparent specific gravity 1.604.

Volumetric proportions were assumed and corresponding weight proportions computed for mortar of normal consistency. The results are given in Table I. The results of the tests are given in Table II.

The decrease in strength of the mortar after an excess of cement paste is used, is not typical of all sands tested. The maximum density was uniformly in the vicinity of the percentage of voids, and this mixture generally showed minimum cost per unit of strength.

Mr. Kitts. To further support my conclusion that maximum density gives minimum cost per unit of strength, I will refer to Prof. M. O. Withey's very thorough tests of mortars.[1] Using his Fig. 10, "Ratios of Strength to Cost," weight proportions, for "Proportion of Cement Paste ÷ Proportion of Voids = Unity," are computed and compared with Professor Withey's weight proportions showing minimum cost per unit of strength in Table III.

It will be more interesting to compare the author's figures with Professor Withey's chart and to note that the varying proportions of silt in no way affected the agreement.

The writer is convinced that mathematical and physical science may be applied to the proportioning of concrete aggregates with results of real and practical value.

[1] "Tests of Mortars made from Wisconsin Aggregates," M. O. Withey, *Proceedings*, Am. Soc. Test. Mats., Vol. XIII, pp. 834–857 (1913).

THE MICROSTRUCTURE OF CONCRETES.

By Nathan C. Johnson.

SUMMARY.

In the investigation of concretes as outlined in this paper, the general nature of concretes and the causes of disintegrations are studied and determined by microscopic examination. In such work, developed structures on the polished surfaces of opaque specimens are chosen in preference to their sections on the ground that the preparation of the latter necessitates the loss of the information sought, in a majority of cases.

The results so obtained indicate the underlying causes for the low strength of concrete produced by the best of materials, largely because of the considerable presence in the mass of occluded air. They also indicate secondary disintegrating actions, aside from such chemical actions as may directly attack the cement, as having a greater importance than is commonly ascribed to them, in concretes exposed to either salt or to fresh water. It is further shown that but a small percentage of the total cement in concrete is hydrated, the greater part lying practically inert in the mass. Lastly, the influence of surface tension of water as a prime factor in the making of good concrete is demonstrated.

No claim is made that microscopic examination is a cure-all for the ills that affect concrete, but in conjunction with other methods, it is held to be highly advantageous and desirable, especially in the examination of sand and stone and in the determination of causes of failure which would otherwise be impossible of exact explanation.

THE MICROSTRUCTURE OF CONCRETES.

By Nathan C. Johnson.

Introduction.

In the *Engineering News* of October 30, 1913, appeared an editorial, reading in part as follows:

> "No subject has attracted greater attention from engineers interested in concrete construction during the past two or three years than the question of the durability of concrete. For at least a generation, it has been universally believed by the engineering profession that good concrete, properly made with sound cement, produced an artificial stone which was at least as durable on exposure to the weather as a sound natural stone. The fact that a very large amount of concrete laid during the past ten years and exposed to the weather and to percolation, in retaining walls and elsewhere, is now showing unmistakable signs of great deterioration, gives engineers much anxiety.
>
> . . . . "If concrete is to be exposed to the weather, the less porous it can be made, the better. Other troubles that have been experienced with concrete may likewise be traced to the porosity of the material. We may refer especially to the failures which have taken place in some docks, where sea water percolated through the concrete and gradually weakened it; and also the well-known difficulties with sea walls of concrete between high and low tides in climates of heavy frost, where the freezing of the water in the pores brings about rapid deterioration."

Whether the deteriorating actions described in this editorial are correctly held to be responsible for the condition of concretes exposed to the elements, these expressions may be said to fairly voice the consensus of present engineering opinion with regard to the durability of concrete; and this being so, there is an inferential distrust of it for certain classes of construction, which is uncomfortable to contemplate. Concrete is a young material, so far as widespread use is concerned, yet its present position is preeminent. The responsibility daily placed upon it, both in lives and property, is of the greatest; and if any general feeling of distrust of its reliability were to occur, the result would be unfortunate in the extreme. Believing, therefore, that any method of investigation which promises any relief from present limitations needs no justification, the results of microscopic

examinations of commercial concretes taken from structures exposed to varying conditions of service are here set forth.

With respect to product, the present position of the concrete industry is not unlike that of the steel industry some twenty years ago. Even with the best of raw materials and supposedly uniform and standard conditions of manufacture, the quality of concrete is always uncertain and generally deficient. The making of concrete is hap-hazard, in that it is impossible to predict results; and as the defects of concrete are hidden, so far as outward examination can reveal them, failure or deterioration in service is usually the first intimation of inner structural weakness. But to the aid of the steel industry came structural examination with the microscope and the development of the science of metallography. If the same means can, either directly or indirectly, work to the same end with concretes, so that the strength of concrete can be predetermined, with full reliance placed on its quality and endurance, its uses will be extended a hundred fold and its right to a preeminent position as a structural material established beyond cavil. It is the author's belief that this will eventually prove to be the case.

Unlike steel, however, all the constituent materials of concrete do not lose their identities in the mass. Any concrete, therefore, is dependent for its mass properties upon the sum of a number of individual properties, and any critical studies for the determination of cause and effect necessitates consideration of each of these.

CONSTITUENTS OF CONCRETE.

Four substances enter primarily into concrete, namely, stone, sand, cement and water. After the chemical union of cement and water in the forming of the binding substance, a new substance with new properties, in addition to the other substances and their properties, complicates the properties of the mass. These substances must therefore be first studied individually and afterward collectively in order to understand the mass behavior.

Stone.—The large aggregate, broken stone, or gravel—the latter, water-worn stone—may seem too well known to warrant

particular study. The word "stone" is synonymous in the popular mind with hardness, durability and resistance to weather conditions, yet all stones do disintegrate, with greater or less facility, according to their composition and structure. Weathered rocks give visual evidence of this. The soil which the farmer tills is disintegrated rock; and the sand grains that form the fine aggregate of concrete are but the component particles of stone which has broken down from a variety of causes, which geologists know well. In utilizing these materials in concrete, therefore, due regard must be had to these matters.

Some stones are indeed natural concretes. Sandstone is plainly representative of such, as can be seen from Fig. 1, which shows the structure of a very small piece of a coarse sandstone (Medina sandstone) at a magnification of 40 diameters, while in Fig. 2 the same field is shown at a magnification of 80 diameters; and in Fig. 3 at 200 diameters. It is very evident that the stone is built up of tiny particles, which we know to be quartz, cemented together by either iron or alumina or carbonates. In each of these photographs are seen black spots, in the cementing material between the quartz grains. These spots are the tiny holes or pores, which make sandstone porous; and it is significant of the analogy which these stones bear to artificial concretes that the durability of the stone and its resistance to weathering are in inverse ratio to its porosity. This is to be expected, when it is considered that not only do such pores provide spaces for the retention of water, which will act disruptively if crystallized by freezing, but further that they provide entrance channels for solvents which will attack the cementing material and leave the quartz grains without bond. Such action in mass, with the reduction of concretionary sandstone to a sand deposit, is shown in Fig. 4.

Sandstone, however, is not the principal reliance for concrete aggregate. Trap, limestone and granite take precedence over it, so that the structure exhibited by these rocks will be of interest.

In Fig. 5 is shown a coarsely crystallin limestone from Palatine Bridge, N. Y. Though less analogous than the sandstone to the structure of artificial concretes, it is not dissimilar in its arrangement of constituent particles. In Fig. 6 is shown

FIG. 1.—Magnification, × 40.

FIG. 2.—Magnification, × 80.

FIG. 3.—Magnification, × 200.

COARSE SANDSTONE, MEDINA.

the structure of trap rock, with its compact, but heterogeneous arrangement of minerals (Nyack Trap); and in Fig. 7 is shown the structure of Northville, N. Y., granite, with its dissimilar materials in large plates, strongly veined and apparently possessing cleavages in its minute portions which would need only release from the binding actions of adjacent materials to be made outwardly evident. This finds expression in one way by the behavior of these materials under extreme heat, as in the fire

FIG. 4.—Concretionary Sandstone, Weathered by Solution and Wind Action. Snake Island, near Nanaimo, B. C.[1]

at the Edison plant at West Orange, N. J., whether or not they have had their identities concealed by incorporation in a mass of concrete. Under heat we should expect these dissimilar constituents to expand to different degrees and since the crystals are arranged helter-skelter, with axial expansion in one case normal to axial expansion in another, disruption is likely to occur, if the degree of heat is sufficient. With limestone, of course, somewhat different actions take place, with possible

[1] From "Engineering Geology," by Ries and Watson.

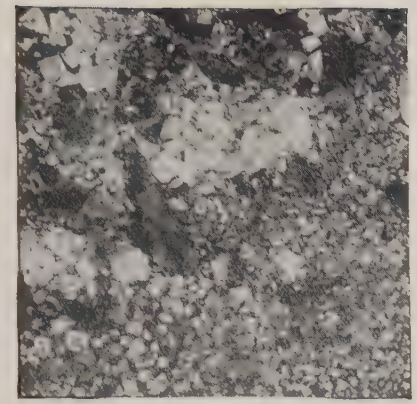


FIG. 5.—Limestone, Palatine Bridge (× 50).

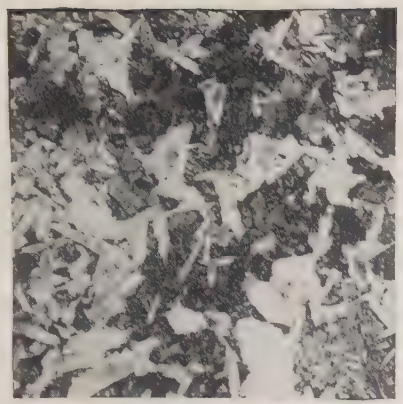


FIG. 6.—Diabase, Nyack (× 50).

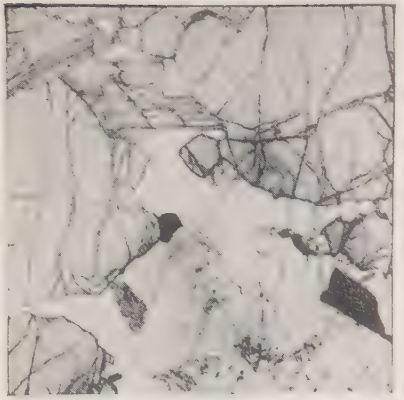


FIG. 7.—Granite, Northville, N. Y. (× 50).

change in chemical composition and resultant disintegration, or spalling; and since the sand grains are in many cases but smaller portions of like materials, it is reasonable to expect the same actions to obtain to a relatively great extent with them as with the larger aggregate. Such actions, multiplied millions of times, in the sand grains and thousands of times in the pieces of stone carried by the concrete of a structural member, such as a column or a beam, must necessarily result in outward spalling or cracking. Concrete is limited in its properties by the properties of the materials of which it is composed; and there is no need

FIG. 8.—Water Drops from Atomizer (× 20).

to apologize for any behavior of concrete under extreme conditions, such as obtained in the Edison fire, when the behavior of these constituent materials under like conditions must, by their nature and structure, be the same.

Sand.—From the analogy as to composition and structure drawn between sand and stone, it follows that the properties of sand as regards strength, compressive or shearing, must closely follow those of stone. Indeed, it is certain that the strength of sand per unit area is not less, and that it is probably greater, than the strength per unit area of stone, since sand is disintegrated stone. Without cementitious material, they have

lost the weak portion of the combination—the cement—and possess the strength of the pure material.

Cement.—The third ingredient of concrete is cement. Briefly, this is a more or less definite compound, containing in varying amounts and combined in devious ways a number of chemical radicals, CaO, MgO, SiO_2, Al_2O_3, Fe_2O_3, SO_3, H_2O and so on, some of these compounds due to the cement materials themselves and others to the secondary addition of gypsum to control the setting. For the present this product will be assumed as satisfactory from the chemical standpoint and no examination made of its microstructure, previous to its admixture with water.

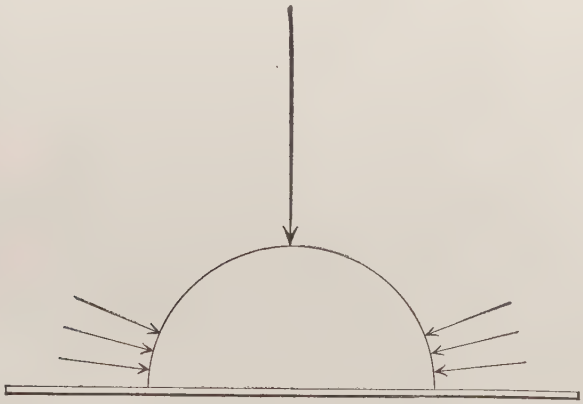


FIG. 9.—Diagram of Section of Water Drop and the Forces Acting Thereon.

Water.—The fourth ingredient of concrete is water, and despite its familiarity it deserves particular study. Assuming its freedom from substances injurious to concrete, its physical properties have a most important bearing on the production of concrete; and of these physical properties, that peculiarity known as "surface tension" is of special interest.

Briefly stated, surface tension is that balance of molecular forces in water which finds expression in such ways as the formation of spherical particles, as in rain drops. This holds true even in the formation of very minute particles, as in the spray from an atomizer, shown in Fig. 8, where the spray has been photographed after falling on a glass microscope slide. It is

evident that each of these particles is a sphere, or at least that each tends to assume the spherical form. If a section through one of these drops were taken, it is evident that since the center of the drop is higher than the other portions, as in Fig. 9, to maintain equilibrium and resist the action of gravity, there must be inward components acting at the sides. So strong are these components that it seems as if there must actually be a tough skin on the water drop, holding the inner portions in place and resisting its spreading over the surface on which it lies. This finds every-day expression in the reluctance of water to pass over a dry surface, such as that of sand or stone; and it also is the cause of the compacting of sands of varying degrees of fineness with certain quantities of water; and their dispersion, or increase of volume, with other quantities. It is evident on these accounts alone, that the surface tension of water is a factor to be reckoned with in making concrete, for not only will it affect the thoroughness with which the distribution of the cement is effected over the surface of the aggregates, but it will further influence the quantities of the materials contained in a struck measure, which is essentially the manner in which the materials for concrete are proportioned. And as will be pointed out later, it also has a most pronounced effect on the thoroughness of the action permitted between cement and water; and as this action produces the binding substance which holds the sand and stone together, it is a prime factor in its collective actions on the strength and durability of concrete. Although seemingly elementary, a thorough understanding of these matters is essential to a comprehension of the formations found by microexamination of concretes.

Physical and Chemical Reactions.—Briefly then, the mixing of concrete involves primarily, a number of physical actions and secondarily, a combination of physical and chemical actions. First of all are the physical actions of wetting three dry substances—stone, sand and cement—with water; and following this is the formation of a binder through the chemical union of cement and water, with sequent physical action in the spread of this new substance over the surface of the sand grains and stone particles, so that they will be strongly held together. The exact actions attendant upon the production of this binder

are somewhat obscure, but they are known to take place in two stages. In the first, the mass loses its plasticity and becomes more or less friable. In the second, consolidation takes place, the mass increasing in hardness until a stony texture is obtained. The first stage is known as "setting;" and the second stage, as "hardening."

It follows from the complex character of its composition, that the reactions involved in the setting and hardening of Portland cement are themselves complex. It seems probable,

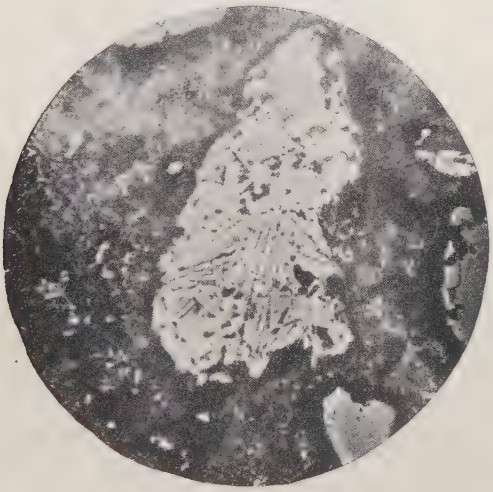


FIG. 10.—Crystals Obtained from Breathing on Clinker; Inside Wall, 138th St. and East River, New York (× 150).

however, that the reactions of setting involve the formation of supersaturated solutions and the deposition therefrom of close-knitted, interlacing crystals of various substances, perhaps such as those shown in Fig. 10, which were formed by breathing on the polished surface of a piece of cement clinker, while the slower reactions of hardening consist partly in the formation of similar interlacing crystallin products, but more especially in the production of a colloidal, or amorphous "gel," or glue, probably colloidal calcium hydrosilicate, with its gradual desiccation.

Obviously, for the proper employment of this binding

substance in concrete, there must be (1) thorough reaction between the cement powder and water; (2) thorough contact of the resultant binder with the sand and stone; and (3) a maximum quantity of sand and stone in any given volume, with consequent minimum of binder, since not only is the binder of indefinite composition and properties but it is further the most expensive ingredient of the concrete, inherently the weakest under stress and in addition, the most subject to injurious attack.

STRUCTURE OF CONCRETE.

Taking up these questions in reverse order, consider the actual structures of concretes, to determine as far as possible the causes of their known deficiencies.

To obtain the minimum quantity of binder and the maximum of stone in a given volume, it is obviously necessary to so grade the aggregates that the smaller sizes shall progressively fill the spaces, or voids, between the larger. Commonly, either arbitrary proportions are used, or else void determinations are made on each material, with an apportioning of each such that the spaces of one will supposedly be filled by the next smaller size. It may be well to investigate the result of such efforts.

In Fig. 11 is shown a piece of laboratory-mixed concrete, proportioned 1 : 2 : 4 arbitrarily, although the void determinations were not far from these same figures. But had the proportioning been done strictly on the basis of void determinations, the same general conditions in the mass would obtain, which are now to be examined.

It is first to be noted that in this fragment of concrete, the pieces of stone are not at any visible point in contact as they would have been in making a void determination, and although in getting this surface a considerable depth was ground away, with frequent examinations of the surface, yet at no time was there found any contact between them. Evidently, if voids were to be determined on these large materials in their present positions, the results would be quite different from those obtained when they were in contact; and if the same relative degree of separation is to hold for the finer materials as well, there would be grave reason to distrust proportioning on the basis of voids.

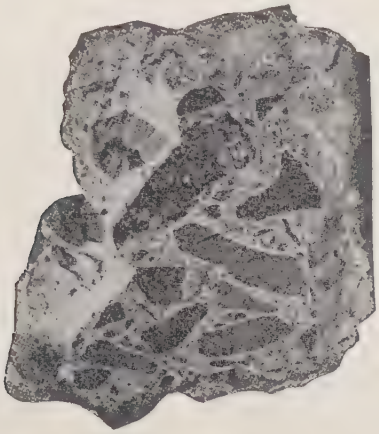


FIG. 11.—$\frac{1}{5}$ Size.

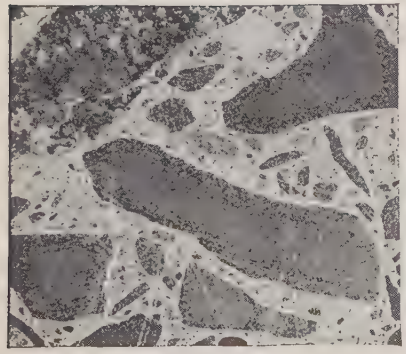


FIG. 12.—$\frac{2}{5}$ Size.

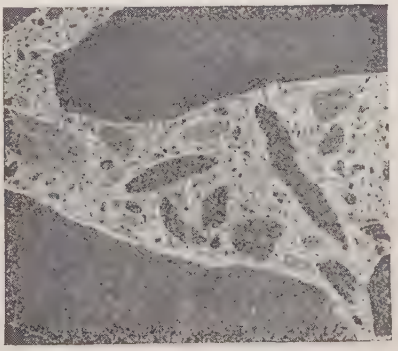


FIG. 13.—Magnification, × $\frac{4}{5}$.

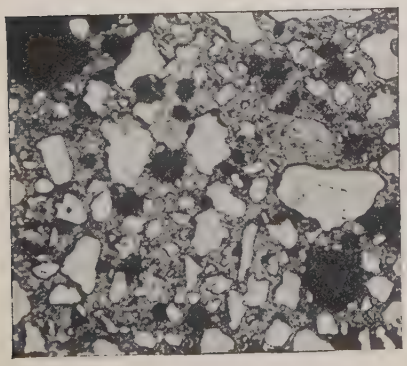


FIG. 14—Magnification, × 10.

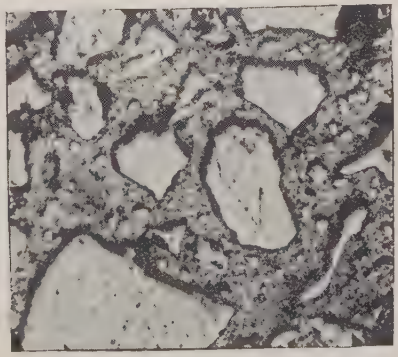
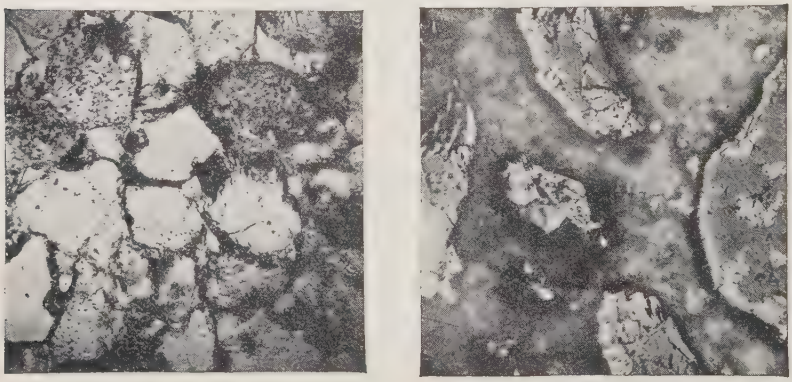


FIG. 15.—Magnification, × 30.

LABORATORY-MIXED, 1 : 2 : 4 CONCRETE.

To determine this point, it may be well to enlarge the surface under examination to different degrees. Fig. 12 shows this surface twice its former size, making visible some of the smaller particles, with the same relative dispersion so far as can be seen. In Fig. 13 the surface is magnified four times its first size, with these features even more evident. In Fig. 14 it is shown magnified 10 times, with color reversal of the various constituents as seen in the photograph, due to the change from oblique to vertically reflected light. Fig. 15 shows the surface magnified 30 times, the area seen in each case being restricted



(*a*) Medina Sandstone (× 115). (*b*) Artificial Concrete. Coaling Plant Pier, Pearl Harbor, Hawaii, H. I. (×115).

FIG. 16.

as the square of the magnification. But in each of these, it is evident that the characteristic conditions noted with the large aggregate obtain with even the smallest particles; that each piece has floated apart from its neighbors to an extent not entering at all into the initial calculations; and that the quantity of binder necessitated by this dispersion is much greater than was planned for, with a proportionate increase in expense, liability to deterioration and lowering of the strength, since cement is inherently the weakest element of the combination in resistance to stress.

To refresh the memory and to draw an instructive compari-

son, Fig. 16 shows, side by side, the structure of the ideal concrete (natural stone) and that of an artificial though excellent concrete (Pearl Harbor Coaling Plant Pier, Hawaii). The strength of the first is from 11,000 to 16,000 lb. per sq. in., while that of the second is from 2000 to 3000 lb. per sq. in. Unless the inference is mistaken, increased quantity of cement in a concrete not only means increased expense, but, further, the quality of the concrete is thereby weakened, provided proper methods

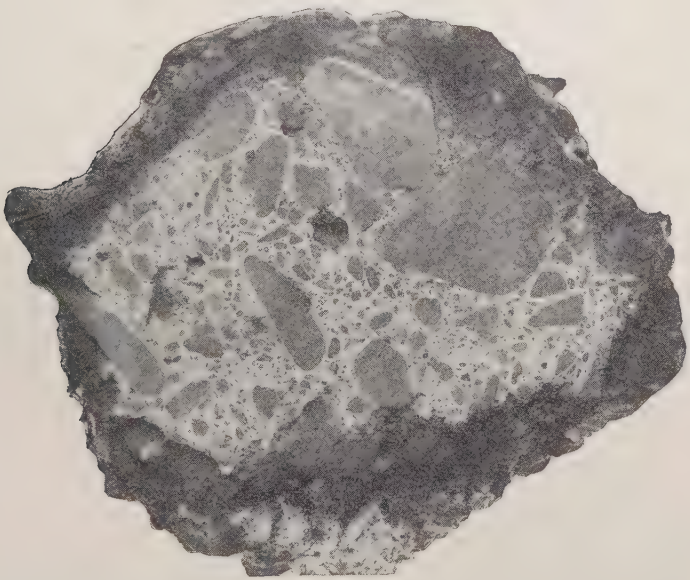


FIG. 17.—Field Concrete, 1 : 2 : 4. Wing Wall, Beebe Lake Dam (⅓ size).

could be applied to its manufacture, so that the compacting of aggregates could be assured.

In the average field concrete, these same conditions of dispersion obtain to an even greater degree. This is made evident in Fig. 17, which shows at one-third size, a sectioned field concrete, made 1:2:4 from gravel, sand and cement (Wing wall, Beebe Lake Dam, Ithaca, N. Y.). In Fig. 18 also is shown a portion of the same surface, at a magnification of 10 diameters, in which it is evident that the same conditions of

dispersion hold for the fine aggregate of this concrete, just as they did in the laboratory concrete before examined.

But not only does Fig. 18 show this wide dispersion. It also reveals lack of contact between aggregate and binder—the second fundamental necessity in a good concrete. In various portions of the surface will be seen places where there is nothing but holes; and in many cases, these holes, or voids, lie adjacent to the sand and stone, precluding by their presence any contact between the aggregate and the binder. This forces upon the attention the existence in appreciable quantities of an unsuspected substance in concrete, namely, air. This must necessarily

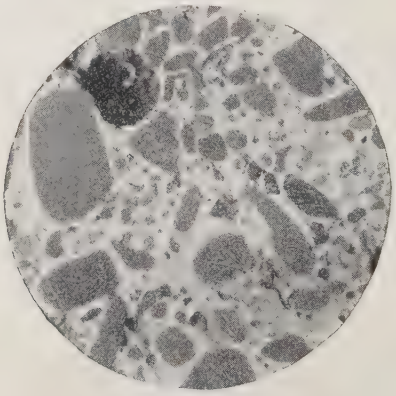


FIG. 18.—Same Surface as Fig. 17 (× 10).

be wholly injurious, since it possesses no properties which could in any way be of advantage to concrete, and reasoning from the analogy of natural stone, it may be highly injurious, since it may form in the mass spaces for the reception of disintegrating substances, or passages for the introduction of solvents into the mass, or both.

But considering first the weakening effect of such occluded air in concrete, it will help to an appreciation of its value to consider Fig. 19, in which are assembled 9 photomicrographs taken in a restricted area on the surface before examined. Below these, for the sake of clearness, is a key drawing, showing the sand grains of each photograph in outline, with the air voids in

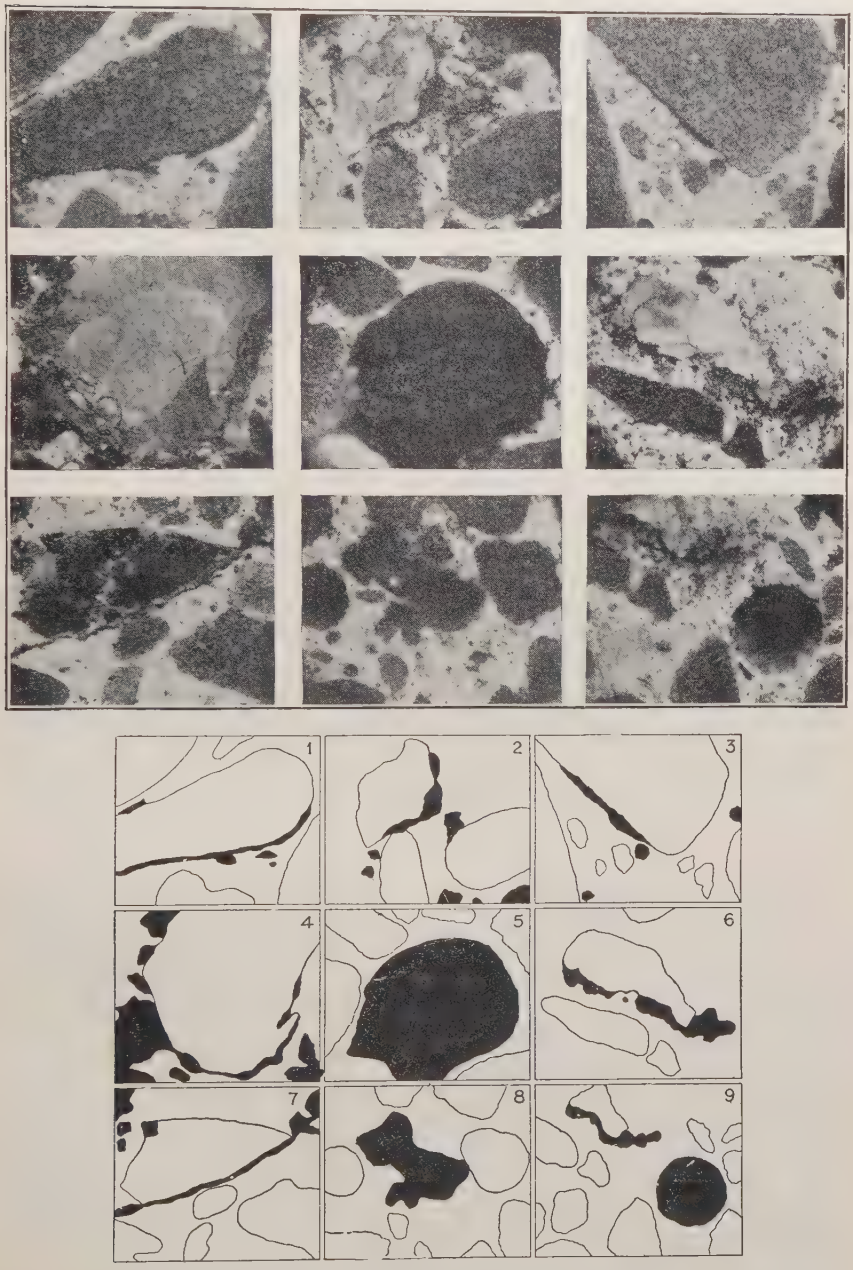


FIG. 19.—Nine Photomicrographs of Surface of Fig. 17. The Key Drawing shows the Sand Grains of each Photograph in Outline, with the Air Voids in Solid Black.

solid black. If a vertical stress is imagined as applied to each of these sand grains, as might very probably be the case were load applied to the mass in which they lie, local failure might be expected in every case, unless each sand grain were of sufficient transverse strength to bridge the air gap. In all probability, however, local failure would result, with other local failures where like conditions exist following in rapid sequence, until the mass failed through such repetition. If these actions were duplicated some hundreds of thousands of times in a test cylinder, for instance, one of the reasons for failure to reach a mass strength even approximating the strength of the aggregate entering into it, could be readily understood.

It is doubtful if the prevalence of occluded air in concrete is appreciated by the majority. To give an appreciation of this, Figs. 20 to 25 are presented.

Fig. 20 is taken from a dock wall at 138th Street and East River, New York Harbor.

Fig. 21 is taken from a wall at the Astoria Athletic Field, New York City.

Fig. 22 is taken from a dock wall at 120th Street and East River, New York Harbor.

Fig. 23 is taken from the Garden Pier at Atlantic City, N. J.

Fig. 24 is taken from the Pennsylvania Railroad Co. ferry piers, Cedar Street and Hudson River, New York Harbor.

Fig. 25 is taken from bulkhead wall, rear of Pier 20, New York Harbor.

Each of these photographs was taken at low power,—15 diameters,— in order to include as much of the surface as possible in the field of the microscope and to show the general characteristics of the concrete. All aggregates are of course sand grains, as even a portion of the surface of a piece of the larger aggregate would completely obscure the field.

There is another consideration applying to the formation of voids, which it is proper to recognize at this juncture. Many of the voids seen in the photographs just referred to, and many of the fissures both in the cement matrix and adjacent to the sand grains, may be due to the use of excess water in mixing and to its evaporation and seepage, subsequent to the setting of the cement rather than to occlusions of air. Every 62.5 lb. of

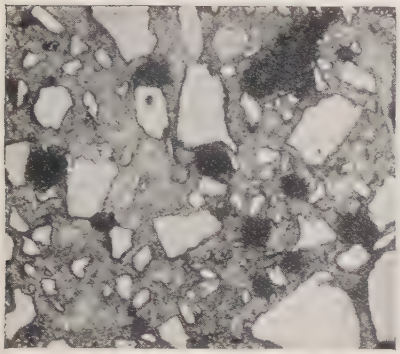


FIG. 20.—From Dock Wall, 138th St. and East River, New York.

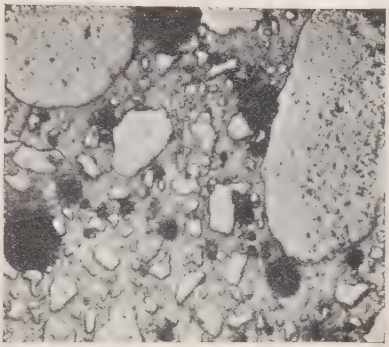


FIG. 21.—From Wall, Astoria Athletic Field, New York.

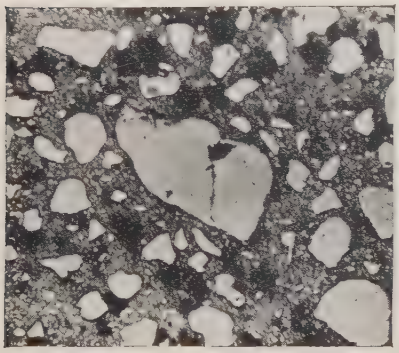


FIG. 22.—From Dock Wall, 120th St. and East River, New York.

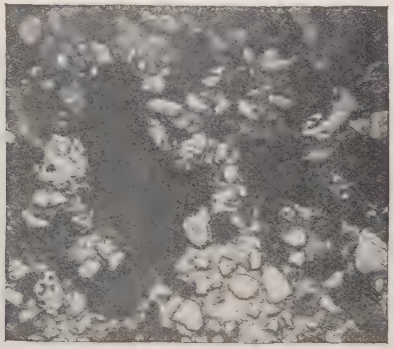


FIG. 23.—From Garden Pier, Atlantic City, N. J.

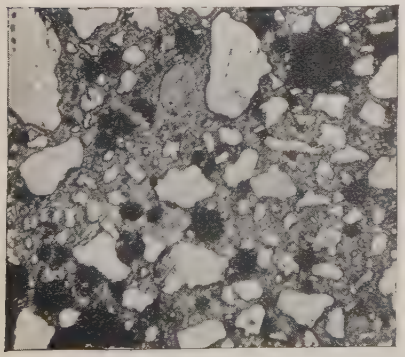


FIG. 24.—From P. R. R. Ferry Piers, Cedar St., New York.

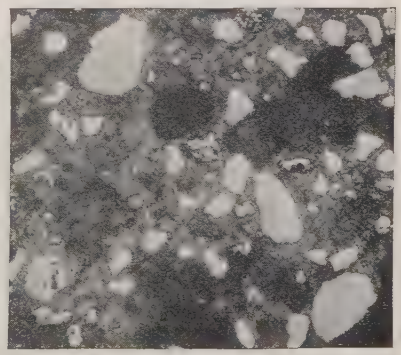


FIG. 25.—From Bulkhead Wall, Rear of Pier 20, New York.

MAGNIFICATION, × 15.

water-weight in concrete occupies 1 cu. ft. of space, and the loss of this amount of water must leave an equivalent amount of voids. But although attributing the voids just seen to excess of water is a matter of conjecture only, unmistakable evidences of shrinkage due to excess water and its loss are frequently found in concretes. Fig. 26 shows, at a magnification of 150 diameters, a single-branch shrinkage crack formed in this way (inside wall, 138th St. & East River, New York), and Fig 27 shows a three-branch crack attributable to the same cause (rear of Pier 20,

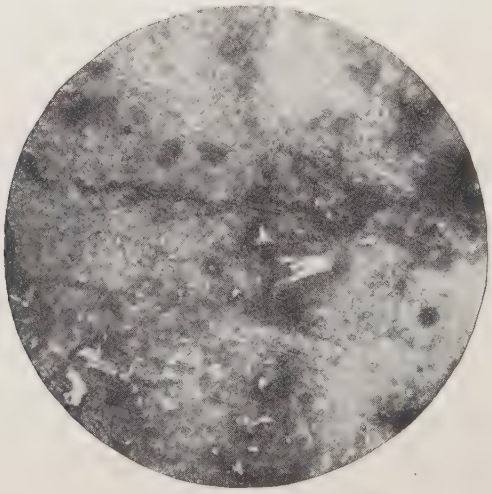


FIG. 26.—Single-Branch Shrinkage Crack (× 150).

East River, New York). These are but two instances out of many at hand.

It may be objected that these microstructures are of little practical importance, or to damn them utterly, that they are "purely academic,"—quoting the ultimate anathema most frequently met with. To prove the truth or lack of truth of such an assertion, attention is directed to Fig. 28, in which is shown the surface of a sectioned 8-in. cylinder of 1:2:4 laboratory concrete, after crushing in a testing machine. To make the features plain, stone surfaces have been blackened, the pores dotted in, and the lines of breakage due to crushing traced with ink. Many of these latter were too fine to follow, but it is

significant as to the effect of these voids, so far as strength is concerned, that the lines of failure run from pore to pore. Further, it is of great significance that these failure lines apparently keep seeking the large aggregate, although none of that which is visible has been crushed. In Fig. 29, the reason is made plain, for in this—a sectioned 1 :2 :5 concrete of the same materials—some of the stones became dislodged in grinding; and it can be seen that where these stones lie, there is a maximum of air voids, close against the surface of the pieces. Remembering the reluctance of water to pass over the surface of stone and the

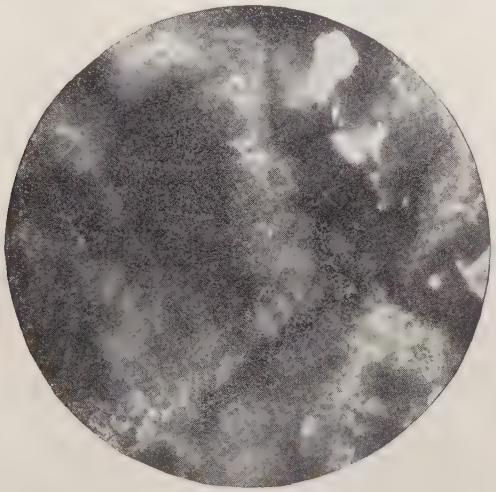


FIG. 27.—Three-Branch Shrinkage Crack (× 150).

tenacity of its own surface, well suited to the retention of a film, or of bubbles of air against the stone and between points of contact between the water and the stone, another effect of the surface tension of mixing water is made evident. And when the prevalence of these air bells in contact with the stone is considered, preventing adequate bedding of the stone in the cement mortar, the bridging of voids by aggregate is seen to obtain in the gross as well as in the microscopic portions of the mass.

Thus far the structure of concretes has been examined only in reduced size, or at low magnifications. Yet even so, there

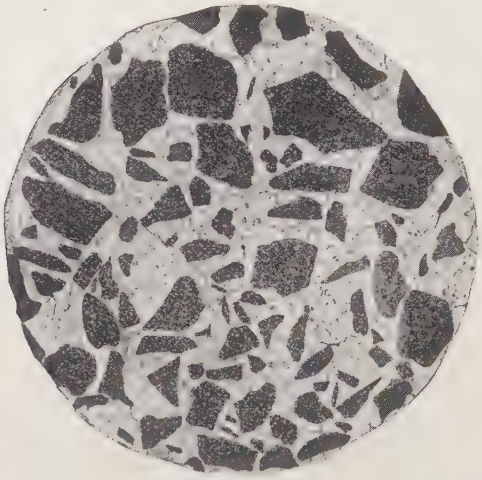


FIG. 28.—Crushed and Sectioned Cylinder of 1 : 2 : 4 Concrete ($\frac{1}{4}$ size).

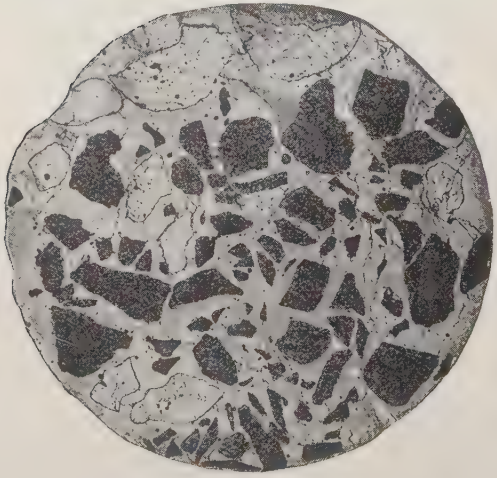


FIG. 29.—Crushed and Sectioned Cylinder of 1 : 2 : 5 Concrete ($\frac{1}{4}$ size).

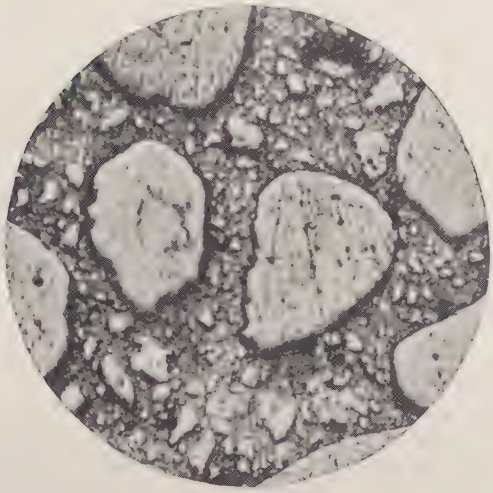


FIG. 30.—Standard Sand Mortar, 1 : 3 (× 50).

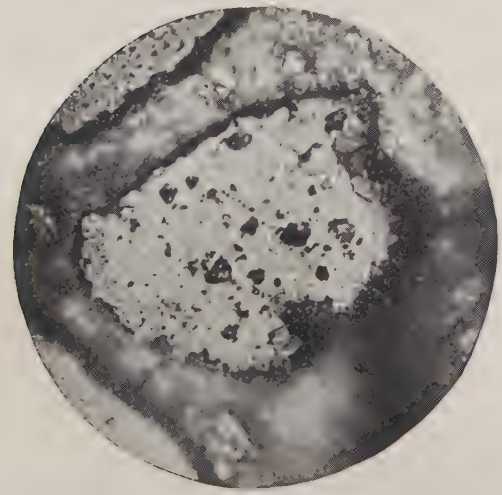


FIG. 31.—Standard Sand Mortar, 1 : 3 (× 200).

have been observed in the sections solid particles which were too small to be sand and too large to be individual particles of cement, nor did they partake of the character of the matrix of hydrated cement. Such a surface is shown at 50 diameters in Fig. 30 (standard sand mortar). Investigation proves these particles to be *unhydrated cement;* and if their prevalence in this mortar briquette, made under standard conditions, is any criterion of their prevalence in concrete, the first condition laid down as one of the three fundamentals of the production of good concrete, that is, thorough reaction between cement and water for the production of the binder, is seen to be violated. Further, through the existence in the mass of this unconverted cement, allowance may have to be made for new and unexpected reactions, if by some means, such as a capillary pore, water were to reach it at some period subsequent to the final setting of the mass.

It may be well to defer consideration of this and related questions for the present, to prove the identity of these lumps as unhydrated cement. In Fig. 31 is shown at 200 diameters a portion of the surface of the standard sand briquette examined before, but at four times the former size. A certain amount of extra polishing has been done, in the course of which softer portions have been cut away leaving the harder portions in relief, or to use the technical expression, the surface has been "developed." In this way the structure of the cement group is made evident. Near the edges of the photographs are seen segments of the standard sand grains, and since in making this briquette only standard sand and cement were used, the inference is fair that the structure shown is that of a group of particles of cement.

It may be objected that this lump is too large to be a single particle of cement, as is the case, and that if it were a group of cement particles, as might be indicated by its appearance, they would hydrate and fall apart. To determine this, recourse must be had to a consideration of the physical actions involved in wetting dry cement with water.

In Fig. 32 is shown, at a magnification of 20 diameters, the appearance of dry cement as it lies on a microscope slide. It is to be noted that its distribution is very uniform. But if water

is added, quite different conditions prevail. In Fig. 33 the water may be seen progressing across the field, but instead of becoming uniformly moistened and remaining in essentially its first position, the fine material is grouped in irregular masses of varying size; and it is plain to be seen that some extraneous force would be required to break up these masses, since they were formed through the action of certain forces which their own inertia was inadequate to resist. Without going to a lengthy demonstration, it will be stated that the operative force in this case is once again the surface tension of the mixing water; that this grouping is but another demonstration of those forces which cause water particles to take spherical form; and that the finer the particles

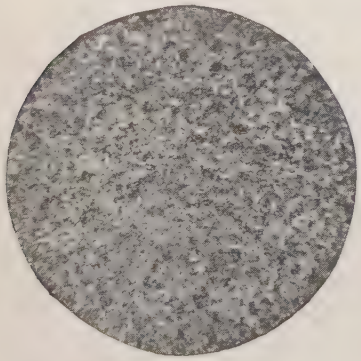


FIG. 32.—Dry Cement (× 20).

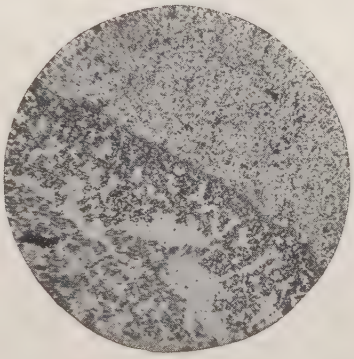


FIG. 33.—Cement Gaging (× 20).

of dry substance are, the more closely may they be grouped. Extreme fineness of grinding of cements might in this way be detrimental to efficient hydration.

But a second set of actions is involved in the formation of these cement groups. In the process of grouping, the particles have become wetted. This wetting has immediately resulted in chemical reaction with the cement, and as the quantity of water remaining between the particles necessarily becomes almost immediately limited, through the closeness of the grouping, a saturated solution of binding product is speedily formed between the particles, with deposition of interlacing crystals therefrom, which operate to hold the particles rigidly in their grouped condition. This action takes place in far shorter time

than is usually required for the production of initial set under usual conditions; and unless mechanical agitations of mixing are sufficient to break down this crystallin bond, they will remain unhydrated except at their outer surfaces, so long as the concrete endures. Further, the bond between these particles is speedily rendered more enduring and secure by secondary colloidal formations, and their desiccation by absorption of water from them by the interior of the mass. These colloidal boundaries form the honeycomb outlines of the formations seen in Fig. 33, the material within them being the unconverted cement particle, of which they form the outer shell. Their appearance as dark boundaries in some photographs and as light boundaries in others is due to focussing on one portion or another of the group.

To determine the extent to which like and other formations existed in commercial concretes, particularly those exposed to the action of various natural waters, samples were collected from different parts of the world, by means of little bags with address and data card attached. Those to whom requests were made for specimens have uniformly shown a most courteous interest and spirit of cooperation, and although it is impossible to render thanks to each personally, it is desired to express sincere appreciation for their helpfulness and the trouble taken to secure the samples desired. The New York Dock Department is deserving of special thanks, for putting at the disposal of the writer a tug boat and crew on successive days, so that samples might be obtained from all the concrete structures in New York Harbor. By one means and another, a very considerable collection has been obtained, representing in their finished state a very substantial expenditure of time, labor and money. Some of the structures found are shown in Figs. 34 to 41, and continued examination is constantly furnishing new food for thought.

HYDRATION OF THE CEMENT.

In Figs. 34 to 41 it is desired particularly to direct attention to the groupings of unhydrated cement. It should be especially remembered that all of these concretes have been constantly submerged for greater or less periods of time, so that any lack

of hydration is not to be attributed to lack of surrounding moisture.

Fig. 34, taken from Pier 2, Halifax, N. S., shows at a magnification of 110 diameters a large group of particles remaining unhydrated, eight months after the concrete was placed.

Fig. 35, at 110 diameters, is taken from pile caps, Staten Island Municipal Ferry, Battery Park, New York City; eight years old.

Fig. 36, at 110 diameters, is taken from 47th Street and East River, New York Harbor; air-cured block for future pier foundation, one year old.

Fig. 37, at 110 diameters, is taken from bulkhead wall, rear of Pier 20, New York Harbor. It shows the same sort of air-cured concrete as that of Fig. 36 after ten years submergence in salt water.

Fig. 38, at 110 diameters, shows another portion of the same specimen as Fig. 37 and exhibits like structure.

Fig. 39, at 110 diameters, is taken from dock wall, 138th Street and East River, New York Harbor; it is a single piece of clinker, probably eutectic, after submergence for 34 years. This specimen furnishes eloquent comment, in comparison to others, of comparative fineness of grinding of former practice as against present practice.

Fig. 40, at 110 diameters, is taken from a bulkhead wall, Newtown and Wahle Creeks, New York Harbor, after four years exposure to water between tides.

Fig. 41, at 110 diameters, is taken from Pearl Harbor Coaling Plant Pier, Hawaii; one year in air.

Like structures can be found in any and every part of specimens taken from different parts of these structures, so that like conditions of hydration must exist throughout all the mass. Further, none of these specimens is over 0.5 cu. in. in volume, so that the quantity of cement remaining unhydrated in each cubic yard of concrete is a matter to ponder upon. The writer has estimated that not over 20 per cent of total cement added to concrete are hydrated, and although others disagree with him in this respect, he has no present reason to change his opinion. But even granting that so much as 50 per cent were actively

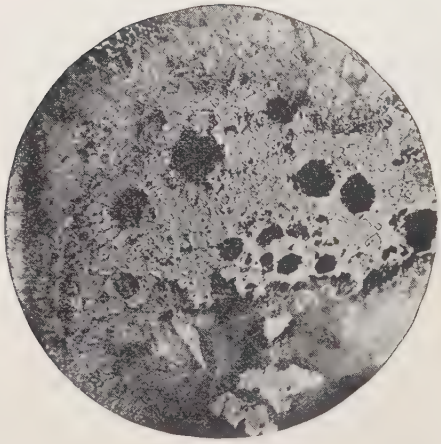


FIG. 34.—From Pier 2, Halifax, N. S.

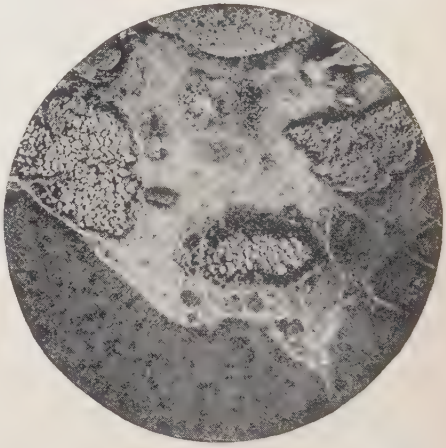


FIG. 35.—From Pile Caps, Staten Island Ferry, New York.

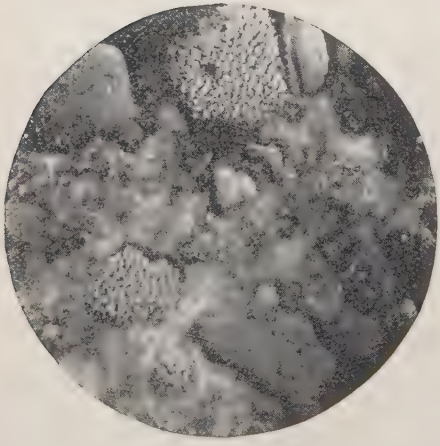


FIG. 36.—From 47th St. and East River, New York.

FIG. 37.—From Pier 20, New York.

GROUPINGS OF UNHYDRATED CEMENT (× 110).

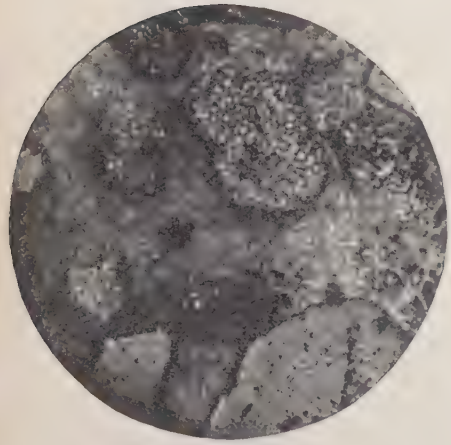


FIG. 38.—From Same Specimen as Fig. 37.

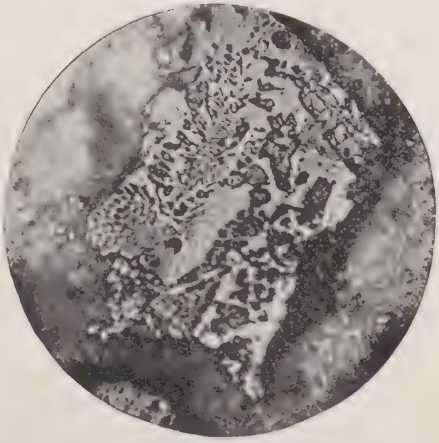


FIG. 39.—From Inside Wall, 138th St. and East River, New York.

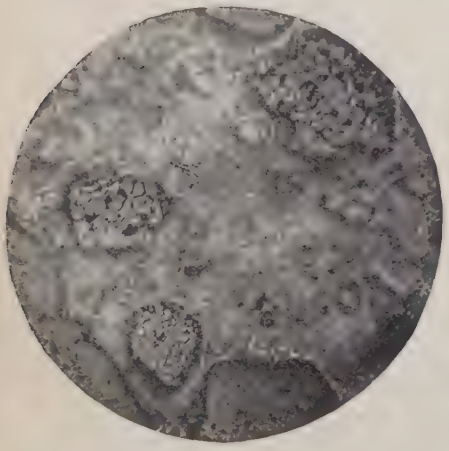


FIG. 40.—From Newtown and Wall Creeks, New York.

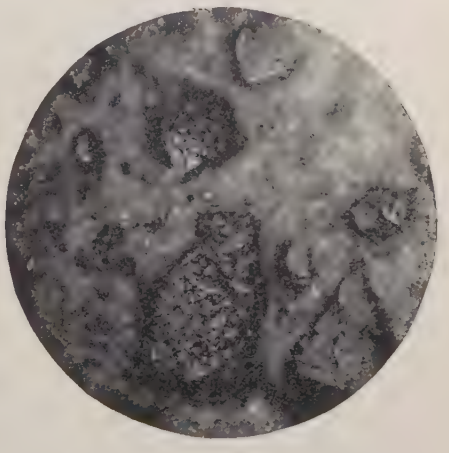


FIG. 41.—From Pearl Harbor Coaling Plant Pier, Hawaii, H. I.

GROUPINGS OF UNHYDRATED CEMENT (× 110).

used, it would be of the greatest advantage to concrete if the percentage of hydration could be increased by some means. The subject of hydration is too vast to permit of adequate treatment in less compass than a complete volume. All that can be done in this paper is to touch upon some of the phases which seem to possess novelty, or upon the results of microexamination that seem to explain and make clear the reasons for phenomena formerly known through other manifestations, but whose exact operation has been conjectural.

Among these latter is the question as to whether a cement particle could become thoroughly hydrated, even under the most advantageous conditions as to access of moisture. It has been held, partly as a result of microexamination by means of polarized light, that there always remained a core of untouched clinker within a colloidal envelope. This view is doubtless correct, and it imposes a limit to hydration entirely aside from the groups of particles previously seen. In the microexamination of opaque surfaces of concretes, this colloidal envelope can also be seen, in the mealy appearance of portions of the matrix. Such a surface is exceedingly difficult to photograph, but in Fig. 42, at 130 diameters, taken from pile caps, Staten Island Ferry, Battery Park, New York Harbor, a fairly clear representation is had. Each of the spots or particles seen in this photograph is in all probability a cement particle, covered with a colloidal, or perhaps colloidal-and-crystallin skin, but remaining quite unhydrated at its center. Consider this condition existing universally throughout the matrix, in addition to the cement-particle groupings before shown, and there is made very evident the reasons why it is possible to regrind, regage and reset an old cement mortar or concrete.

Closely allied on a number of counts with efficiency of hydration is the question of deterioration and failure. It has been previously pointed out that if a mass of cement remains unhydrated in a set concrete and if water is brought to it at a later time through a capillary pore or a fissure, that new compounds with new and indeterminate properties and volumes may be formed in the rigid mass. Proof of this conjecture will be adduced in the following paragraph. In addition to this, the existence of these pores with consequent ready admissibility of

solvent or salt-bearing waters, and preparation for disruptive frost action, is dependent to a large degree upon inefficient hydration, for one of the most valuable properties of hydrating cement is the great volume-filling capacity of the flocculent colloid formed by the union of cement and water. This colloid occupies not only the space taken by the cement, but the space taken by the water as well, so that instead of air or water voids remaining in the mass, the colloid, dense and impervious, occupies

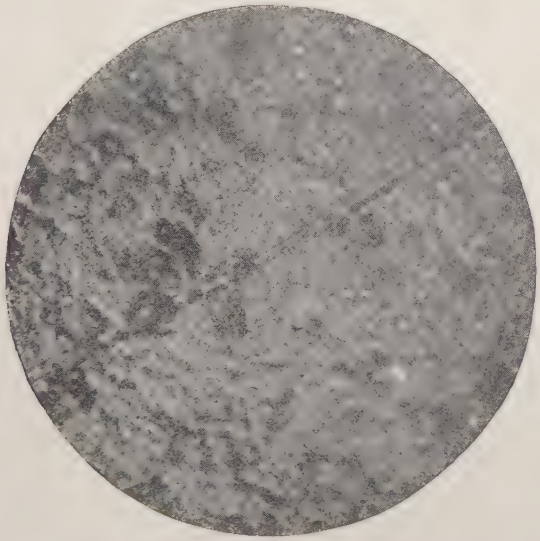


FIG. 42.—Colloidal Envelope on Cement Particle; Staten Island Ferry Pile Caps, New York (× 130).

efficiently all inter-aggregate spaces. This is a highly desirable condition to bring about, on these counts alone.

FAILURE OF SEA-WATER CONCRETES.

The failure of sea-water concretes has probably caused the concrete industry more arduous study, labor and anxiety than any other single trouble. There is yet considerable disagreement among authorities as to the causes of sea-water disintegration, but there seems to be quite general concurrence in the opinion that the formation of calcium sulfo-aluminate by interaction between the sulfates of sea water and the aluminates of

the cement, are in very large measure responsible. This salt, calcium sulfo-aluminate, increases largely in bulk with crystallization, and if such deposition of crystals were to take place in the pores of concrete, disruption by physical action might reasonably be expected. If this disrupting action were repeated numberless times in each of the countless pores existent in the average concrete, serious if not total disintegration might be expected.

If the formation of such an expansive crystallin material were pictured as taking place in the cement matrix of a concrete, there would have to be imagined a gradual straining of the confining material, until rupture took place. It is significant as to the correctness of such an hypothesis that in the cement matrix of all concretes so far examined which show outward signs of disintegration, interior evidences of such strain are found by microscopic study.

In babbitts and bronzes, incipient fracture from stress is evidenced by "shear planes," or "slip bands," where one portion of the material has flowed over the other. In Fig. 43, at 110 diameters, taken from pile caps, Staten Island Ferry Pier, New York Harbor, are shown similar "shear planes" in a sea-water concrete which is noticeably disintegrating. It will be observed that these shear planes are at right angles to the polish scratches on the specimen, so that the lack of identity cannot be questioned. Under visual examination, these show as iridescent bands, changing in color as the focus is changed, as if they went to a considerable depth and at an angle to the plane of the surface examined.

Nor is this an isolated case. This is one of the most characteristic of all the formations found in under-water concretes. A few more examples of the same thing are shown in Figs. 44, 45, and 46.

Fig. 44, at 110 diameters, shows a second sample taken from pile caps, Staten Island Ferry.

Fig. 45, at 110 diameters, shows a third sample taken from pile caps, Staten Island Ferry.

Fig. 46, at 110 diameters, is taken from Pennsylvania Railroad Co. ferry piers, Cedar Street and Hudson River, New York Harbor.

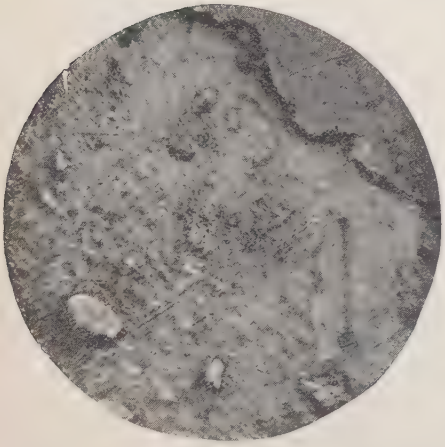


FIG. 43.—From Staten Island Ferry Pile Caps, New York.

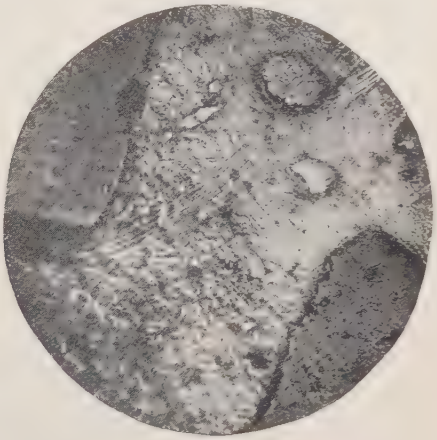


FIG. 44.—Same Location as Fig. 43.

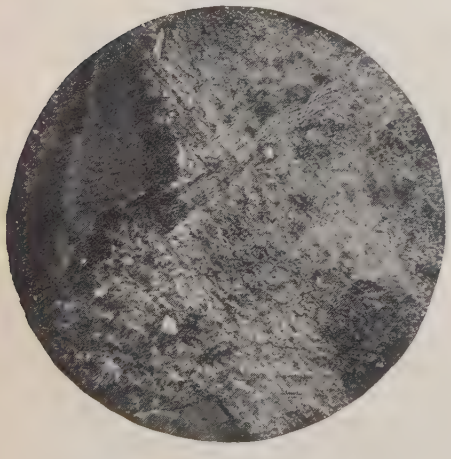


FIG. 45.—Same Location as Fig. 43.

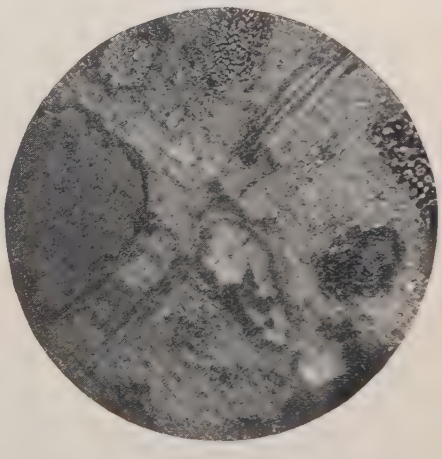


FIG. 46.—From P. R. R. Ferry Piers, New York.

SHEAR PLANES IN UNDER-WATER CONCRETE (× 110).

It is very evident that all are of the same general type and nature and that they indicate a condition of incipient rupture which is not only indicative of tremendous internal strains on the concrete, but also of a possibly dangerous condition of incipient rupture along the planes of these strain bands.

With regard to the latter, it is at least reasonable to infer that any force of a potency capable of causing incipient fracture in the cement matrix would exhibit further manifestations. Assuming that these shear planes are also relief planes for

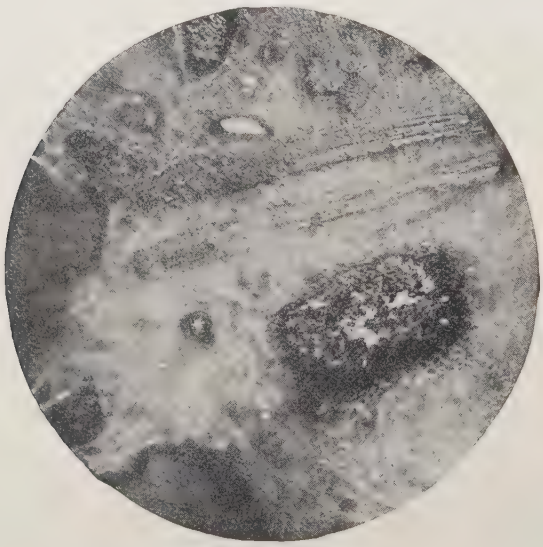


FIG. 47.—Crystallin Intrusions in Shear Planes; Staten Island Ferry Piers, New York (× 130).

tremendous confined crystallin pressures, an extension of the crystallizing substance into such relief planes might be looked for.

In Fig. 47, at 130 diameters, taken from Staten Island Ferry Piers, New York Harbor, such crystallin extension is found. From their character it is evident that these parallel formations, striated and bearing close resemblance to the shear planes before seen, are but intrusions of the crystallizing solutions into the relief fissures first formed. Fig. 48, at 130 diameters, taken from Pennsylvania Railroad Co. Piers, Cedar Street,

New York Harbor, is a further example of the same kind; and the list might readily be extended. Attention is called both in Figs. 47 and 48 and in the photographs preceding, to the proximity of considerable groupings of unhydrated cement to the strain bands and crystallin fillings. It may be incorrect to draw an inference from this proximity, which may be accidental, but it is interesting in view of the suggestion before made as to what might reasonably take place in the set concrete if water were brought into contact with unhydrated cement.

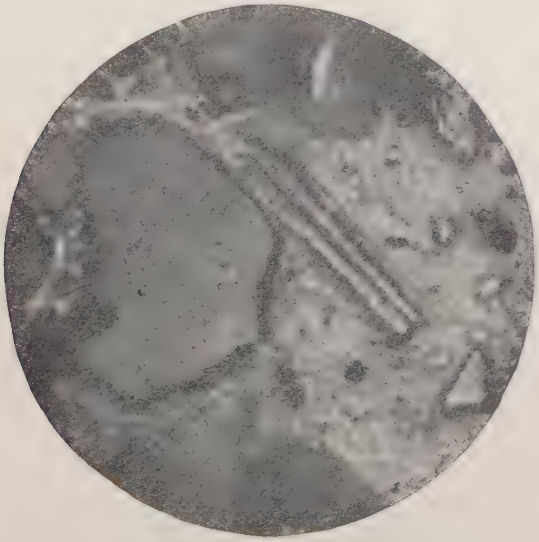


FIG. 48.—Crystallin Intrusions in Shear Planes; P. R. R. Ferry, New York (× 130).

But if these shear planes are actually relief planes for crystallin, expansive formations in the pores of concrete, it is reasonable to expect that these planes would find their origin in a pore or fissure. This is found to be true. It is not always possible to connect the shear plane definitely with any pore or fissure in the plane examined, but in others this can be readily done. In Fig. 49, at 160 diameters, the formation of these relief planes can be seen in radiants from a pore; and in the specimen the hard crystallin filling, different in texture and color from the

cement matrix, can be seen by visual examination almost filling the pore and putting out feelers, or wedges, into the radiating fissures seen in the photograph.

Nor is it reasonable to expect these wedging tentacles to stop with the formation of a slight relief fissure. Such a fissure must necessarily be narrow and as well adapted as was the originating pore to draw up the salt-bearing solutions by capillarity. This would mean, by a repetition of the same actions of capillary attraction and of solution, concentration and crystal-

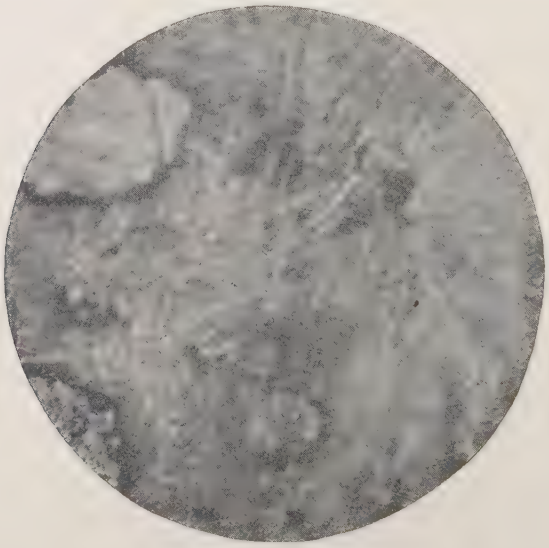


FIG. 49.—Relief Planes Radiating from Pore (× 160).

lization, that the formation of a crystallin intrusive feeler would follow in the relief fissure; and further, that this would grow so long as fresh supplies of solutions could be brought into it.

This being so, larger formations than those previously seen should be apparent. In Fig. 50 such an one is seen, found in the matrix of the concrete from the Steel Pier at Atlantic City, N. J. The photograph was formed by piecing together a succession of photographs of adjoining sections, as the length of this disruptive dike was so great as to preclude its being put on a single plate. In Fig. 51, taken from Beebe Lake Dam, Ithaca, N. Y.,

FIG. 50.—Disruptive Crystallin Dike in "Salt-Water" Concrete from Steel Pier, Atlantic City, N. J. (× 35).

is seen a like formation in a fresh-water concrete; and in Fig. 52 is seen another such dike from another specimen taken from the same structure.

From this it will be seen that these formations are not

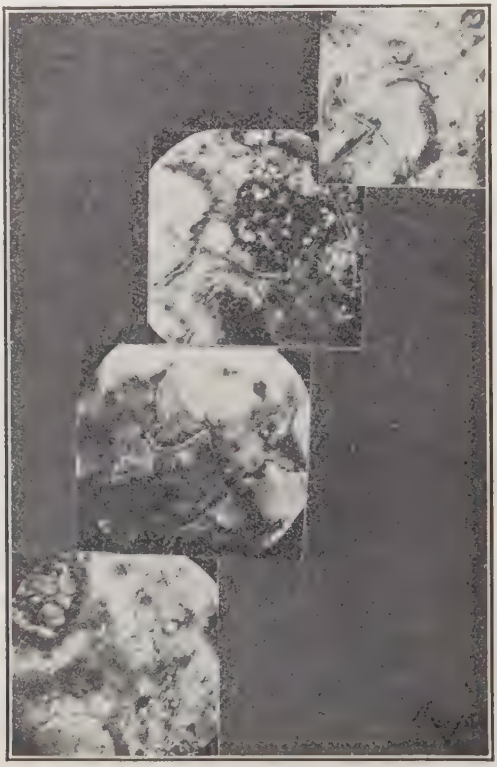


FIG. 51.—Disruptive Crystallin Dike in "Fresh-Water" Concrete from Beebe Lake Dam, Ithaca, N. Y. (× 75).

confined to salt-water disintegrations, and it must be inferred that compounds other than calcium sulfo-aluminate are responsible for some disintegrations. Indeed, it would seem as if this line of reasoning might lead to an exclusion of sea water *per se* as the cause of disintegrations. The type of disintegration here

shown is cancerous in its nature, deriving its malignant substance through solution from the substance destroyed, just as cancer of the human tissues has its sustenance directly from them. If this is true, the problem of making durable concrete for all services is essentially one of making concrete impervious.

Disintegrating actions of a like nature are exhibited in a number of concretes exposed to a wide variety of conditions of service. One of the most common are sidewalks, where ground water is absorbed by capillarity. Figs. 53 to 57, inclusive, show

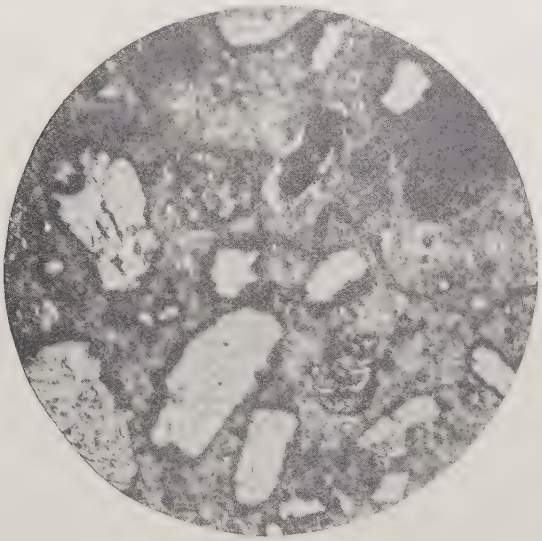


FIG. 52.—Another Dike from Beebe Lake Dam (× 42).

common examples of such. Another common exhibition of them is in retaining or buttress walls exposed to seepage. Fig. 58 (*a*) shows one example of such in a retaining wall, where the crystallin dikes have grown to such size as to be megascopic, sticking out in this case over $\frac{1}{4}$ in. from the face of the wall. In Fig. 58 (*b*) is also shown one cause contributing to this condition, that is, the open porous body of the wall, adapted to admit and hold all the seepage water that touches it. In fact, so common are such conditions that they are accepted as the inevitable, and in the long run, a multiplication of such instances, caused directly

FIG. 53.—Disintegration of Dam due to Crystallin Dikes; Fall Creek Gorge, Ithaca, N. Y.

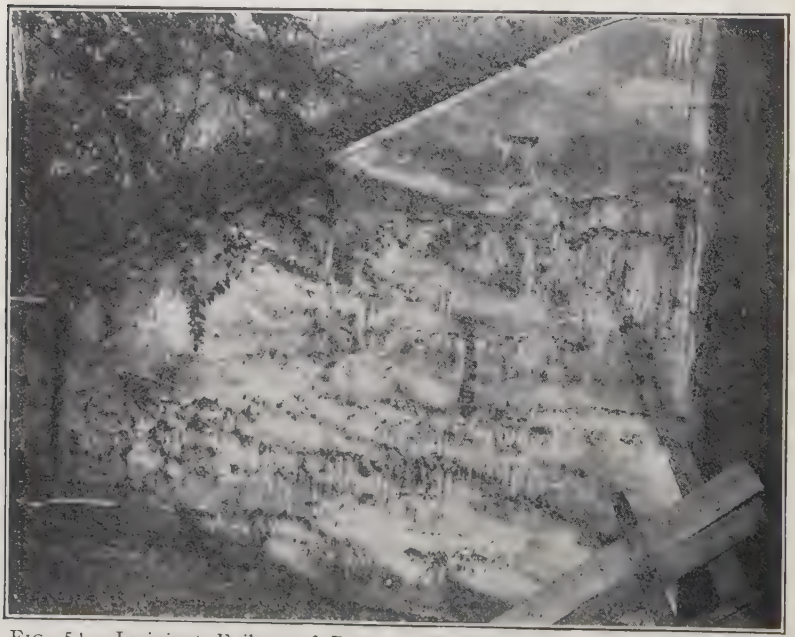


FIG. 54.—Incipient Failure of Buttress Wall due to Extending Crystallin Dikes; Lower State Road, near Ithaca, N. Y.

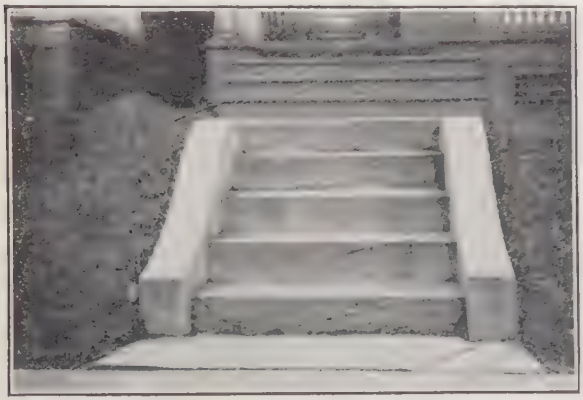


FIG. 55.—Crystallin Disintegrations in Steps.

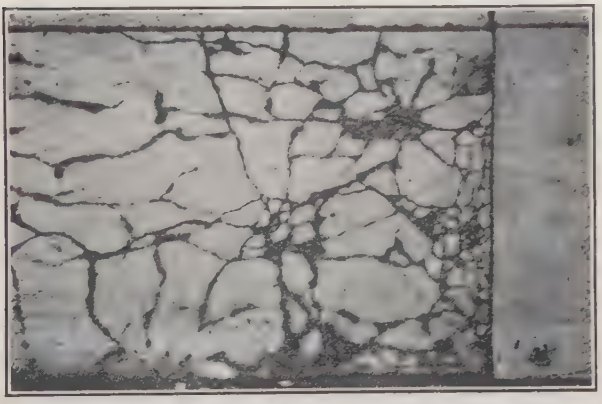


FIG. 56.—Crystallin Disintegrations in Sidewalks.

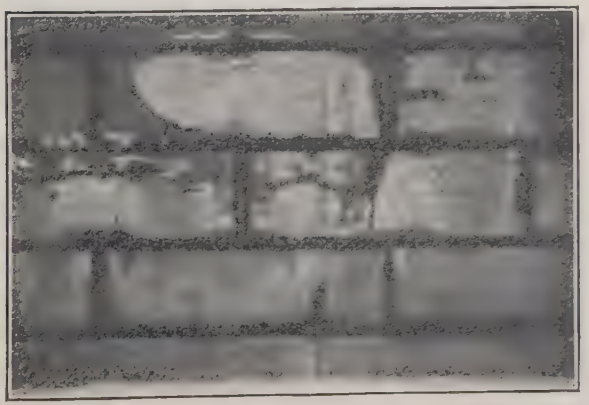
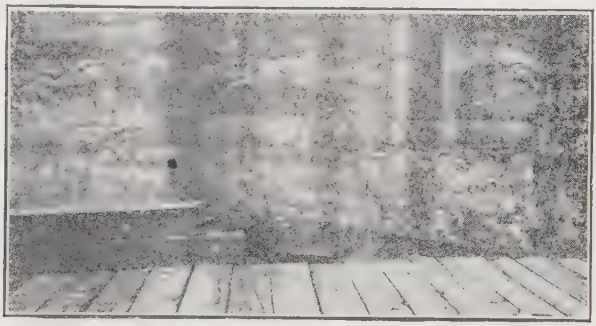


FIG. 57.—Crystallin Disintegrations in Retaining Wall.

by careless workmanship and lack of understanding and knowledge, are going to be factors in creating distrust of concrete in the popular mind, that will do a vast amount of damage to the concrete industry.

It is impossible in the bounds of this paper to go into the very important subject of increasing hydration by one means



(*a*)

(*b*)

FIG. 58.—Crystallin Dikes Disrupting Defective Bulkhead Wall; North Pittston Station, L. & W. V. R. R.

and another. Results already published, available to all who desire to learn of them, have indicated that much can be accomplished through lowering the surface tension of the mixing water by one means or another. These results, however, are not conclusive by any means. They are but tentative, and although

it was expected to have further results available at this time for presentation to the Society, this expectation was not realized through discontinuance of experimentation. This phase of the question, therefore, will be left for discussion until another time.

A word of explanation and caution is not misplaced here. In this paper, defective concretes and causes of failure have been dealt with almost exclusively. This has been done because such concretes are those needing attention and study. Perfect concretes take care of themselves, and they are chiefly of interest in a critical study as forming bases of comparison. With this clearly understood, it is believed that there will be little cause for misinterpreting the attitude of the writer towards concrete as a material for general construction purposes.

DISCUSSION.

Mr. Smith. MR. E. B. SMITH.—In connection with the question of hydration of the cement particles, I should like to mention a few experiments which I have made to determine the effect of vibrations on the strength of mortars and concretes during the period of plasticity. The mortar or concrete was mixed in the usual manner, then immediately placed on a board which was vibrated horizontally, by means of a small motor attachment, at the rate of about 800 vibrations per minute. The amplitude of the vibrations was about $\frac{1}{16}$ in. The vibrations were continuous until about the time of final set. The ordinary tension briquettes were treated in this manner, and also some 6 by 6-in. compression cylinders of concrete. Upon testing for strength after 7 days, it was found that in every case the strength was greater than that of the specimens which had not been vibrated. The average increase in both tension and compression specimens was about 50 per cent, and in a few cases was as high as 98 per cent. An examination under the microscope showed that the colloidal matrix, as I would term it, was very largely increased. I would say that the cement particles were better hydrated by virtue of the mechanical action of vibration, which served to rupture the colloidal envelope and allow the water to come into contact with the cement particle again, this being a continuous process until the mass became too hard to be affected by the vibrations. The microscopic examinations also showed that the vibrated specimens were more dense and remarkably free from voids and air bubbles.

Mr. Lesley. MR. ROBERT W. LESLEY.—Mr. Chairman, the last speaker has just described some experiments of his in vibrating concrete. The vibration of concrete in a very large and practical way has been done in England and Belgium for some time, under certain processes that will be described in the engineering papers. I think it has also been done in this country by what is known as the Jagger process. The nearest resemblance to it would be the motion a man feels on shipboard, where he is turned first one

way and then the other, a sort of double-barreled eccentric motion. The concrete is fed into a machine, and the results just described have been corroborated in a large way by that method. I don't know of any microscopic examination of that concrete, but that might lead to valuable discoveries. Mr. Lesley.

MR. E. S. LARNED.—I believe Mr. Johnson made some experiments with a view of reducing the surface tension and securing a higher percentage of hydration, and I should like to ask him if he would give us a brief outline of what those experiments were and what results he was led to expect. Mr. Larned.

MR. NATHAN C. JOHNSON.—We undertook a series of experiments in lowering the surface tension of the mixing water. Surface tension has a variety of manifestations. The simplest, perhaps, is the formation of a spherical drop. As illustrated in the paper, there must be a very strong action to hold that drop in a spherical shape; it must at least resist gravity. Another manifestation is the reluctance of water to wet a dry surface, such as concrete aggregate, resulting in occlusion of air as shown in the photographs. We therefore lowered the surface tension of the water by the addition of various substances to overcome these difficulties. Our experiments were incomplete and inconclusive. We were encouraged, however, to believe that much could be done in this way. They were published as a matter of interest, not as absolute values, and I should like to see further experiments along the same line. The most promising indication, from a commercial standpoint, was the addition of a very small percentage of some sodium soap solution. I regret that these experiments could not be continued, and that test pieces to be broken at longer periods had to be thrown away because of the abandoning of all experimentation. I hope, however, to take up this work again this winter. Mr. Johnson.

MR. R. J. WIG.—I can answer Mr. Larned's question regarding the value of a material for reducing surface tension. Some experiments which we made showed that the addition of alcohol reduced the strength appreciably. Mr. Wig.

MR. LARNED.—I recall in some account of Mr. Johnson's experiments, his statement that hot water to some extent relieves surface tension. I had the privilege, a number of years ago, of making some experiments on the effect of hot water at different Mr. Larned.

Mr. Larned. temperatures upon the setting properties of three kinds of cement: a normal cement, a very slow setting cement, and a flash cement; and recalling very briefly the effect of high temperatures, you may be interested to know that with water as near the boiling point as we could handle it, that is, between the boiler and the slab where we were doing the mixing, with the neutralizing effect of normal temperatures in the cement used, and of course in the trowel, molds and slab, we found that extremely hot water made a flash cement almost normal in set, it affected normal cement very indifferently, and made slow-setting cement, if anything, a little quicker. Now in the hope that perhaps hot water might largely result in lessening the surface tension, I instituted some tests in anticipation of this meeting, through the courtesy of Mr. Sherman, President of the New England Bureau of Tests, of Boston. We made these tests in the belief that if the surface tension were relieved, the lean mixtures would show improved results. We made one series at normal temperature, starting with a mix of 1 : 3, then 1 : 4, and 1 : 5, using Ottawa sand. We then used the same cement with water at 150° F. with the materials themselves heated to 150° F., with the slabs warmed by the application of hot cloths before exposing the mixture, and with the trowel brought to the same temperature, in a series of 1 : 3, 1 : 4 and 1 : 5 to await the 7 and 28-day results. The results were absolutely negative, indicating no relief whatever at these periods. That is, the normal temperatures gave as good results at 7 and 28 days, as the higher temperatures did, in not only the 1 : 3 mix, but also in the 1 : 4 and 1 : 5. I had great hopes that hot water might accomplish something, because I had known it to do so in winter weather when we were dealing with some questionable materials, and particularly with some varieties of sand. I think, perhaps, it might accomplish relief with some sands where perhaps it would not have a noticeable effect on the cement.

I think the points brought out by this microscopic study of concrete are not only of interest, but of extreme value and importance. It seems to me that they indicate the necessity of not only carefully proportioning the aggregate and cement, but of thoroughly mixing it, which in my opinion is commonly neglected. Where specifications have been prepared with a

Mr. Larned.

view of regulating the time of mixing, it seems to me that in some cases the fundamental principles have been absolutely neglected. No attempt should be made, I think, to prescribe specific periods of time, like three-quarters of a minute, or a minute, for mixing. It certainly depends somewhat on the character of the mix and the proportion we are using, whether one minute is sufficient, or whether we need two minutes or three or more. It depends also on the character of work we are doing. If we are aiming at a particularly dense, impervious concrete for adverse exposure, we cannot adopt too much care in the preparation of the concrete. I do not believe that the natural segregation of materials can be effected in any other way than by lessening the amount of water used, and by the resultant prolonged mixing. I believe these two factors are the most important we have to deal with. In the presence of excess water—a thing I have raised my voice against for years it is impossible to prevent segregation. There is then an immediate segregation from the time the materials are brought together, and while they remain in a liquid state, and are agitated more or less by movement over the forms. I made some experiments at one time with a standard cement and Ottawa sand mixture of 1 : 3, using 20 per cent of water (that is, not as fluid as grout), and used a gang-mold, pouring three briquettes with eight molds laid one over the other, and the joints sealed to prevent the escape of water. The idea was to see how the briquette might be affected in a height of 8 in., agitated as we ordinarily work concrete in forms. The mixture was poured and then simply churned with a pencil or little glass rod, and each end of the gang-mold separated by a very fine-gage wire. After standing about four hours, the briquettes were sawed apart, they being then stiff enough to admit of that handling. There was a gradual loss in strength from the top to the bottom; the bottom mixtures were the weakest and remarkably uniform in the gradation of strength, the greatest strength being found at the top. That is the effect we are producing every day in casting our columns, beams and slabs, with *excess* water, that is, we are getting the strength in the wrong place. I think that can only be avoided by a reduced amount of water, and more thorough mixing. If we do that, then we can increase the coarse aggregate and reduce the percentage of matrix, which I think is also a very valuable thing.

Mr. Thompson. MR. SANFORD E. THOMPSON.—Mr. Johnson's paper is extremely interesting, nearly as much in indicating the possibilities of the microscope, as in the actual results obtained. He has presented results which show and emphasize the necessity for first-class materials, thorough mixing, and proper consistency. His specimens, from necessity, however, have been taken mostly at random from different pieces of work in which the history of the concrete was unknown. The conditions of mixing and the materials are indeterminate, and therefore the causes of the defects, whether due to voids, improper proportioning, insufficient mixing, too wet consistency, the character of the sand, or the composition of the cement, cannot be clearly defined. It is most important that, using these methods as a basis, the tests should be continued in a still more scientific manner by taking up one problem after another and making series of tests; making up known pieces of concrete under various conditions, eliminating the variables and investigating one point and then another. Thus, the effect of different methods of mixing can be determined accurately only by making a large number of specimens under different conditions, and then examining them by photographs at different stages to compare the results. The effect of consistency can be investigated in the same way. The effect of fine and coarse sand on the character of the concrete can thus be studied. The effect of impure sands can be looked into. But in order to get really practical results, it must be done in a manner which will take up the variables one by one. To illustrate the necessity for this, there is a reference in the paper to the probable cause for the voids around the grains of aggregate in the specimens of concrete. It is impossible to tell, however, whether these are due to improper mixing, as is indicated by the author, or whether they are due to some peculiarities in the sand—for example, a coating on the grains—which prevents the adhesion of the cement. Such conditions as these may well be studied by the aid of the microscope, and its use along the lines laid down should be an extremely valuable addition to our other methods of investigation and tests of concrete and concrete materials.

Mr. Johnson. MR. JOHNSON.—In reply to Mr. Thompson, I sincerely hope that this investigation may be carried further in the scientific

manner that he advocates. He is unquestionably right that such is the only way to eliminate these troubles. The slides, made from photographs, have been presented as showing the character of concretes as they are to-day, and as they are found in practice. If we can institute a research in a scientific manner and take up those troubles one by one and eliminate them, I think it will be a very great benefit to concrete. As to the voids being caused by coatings on the sand, in my limited experience there have been no coatings of a thickness commensurate with the size of those voids. I presume Mr. Thompson refers to the voids which lie adjacent to the particles of aggregates. It would be a very thick coating indeed which would cause such fissures, and a coating of such thickness would, I am afraid, almost inhibit the setting of the cement. Mr. Johnson.

MR. THOMPSON.—I should like to add one word with reference to this point. Suppose there is a coating on the sand grains: this coating prevents the concrete from hardening properly and so permits the penetration of water. The flow of the water and the weather action naturally tend to act upon the cement and enlarge the void thus produced, so that when photographs of the defective concrete are taken, it may be that the structure is quite different from the original concrete. The appearance of voids, therefore, does not necessarily prove that the same voids were present when the concrete was first mixed. Mr. Thompso

GENL. W. H. BIXBY.— A word was spoken a moment ago in favor of less water. Now every time I hear less water mentioned, I always feel as if I ought to say something in favor of more water, remembering that local circumstances govern somewhat the question of whether concrete is to be mixed more fluid or more dry. About 17 years ago, I was on the upper Ohio River when they were building some locks and dams, and among the pieces of construction was a lock recess, parts of which would be above the water most of the time at all seasons. One section of wall that I particularly have in mind was about 250 ft. long, 10 ft. wide, and 20 ft. high. Its concrete, which was laid in the fall, about October, quite dry, drier than the average, had a chance to get reasonably set before it was frozen. Now I do not think there would have been any trouble particularly with it, if the work had been located in some southern climate, where not subject to Genl. Bixby.

Genl. Bixby. freezing weather, or in any climate where not subject to extremes of water rise and fall; but on the Ohio that particular winter, after the concrete was in, the river went up two or three times in high flood and promptly went down again, with freezing weather between floods. As a result, owing to the dryness of the concrete when laid, it was quite porous, and during the floods, the wall was pretty well soaked with water. When the floods went down the concrete froze, and when spring came, we went into that one particular piece of concrete wall and dug it out with a shovel to the extent of one-third of its thickness on each side of the wall, and of nearly one-half of its height, so that when we got done with the shovel work, there wasn't very much wall left. Now that would not have happened if it had not been for the rapid succession of freezings between the rapid successions of rise and fall of water around the wall. Of course, when we rebuilt the wall, we used better cement, mixed the concrete a little bit wetter than before, and rammed it better, so that it was more solid and less porous; and it was not injured the following winter.

What I want to bring out particularly is the fact that all these questions of quantities and qualities must be considered as relative, and as largely dependent on local conditions; so that what might be good in one place, not liable to thorough soaking and subsequent freezing, may be quite unsuited to another place where the conditions are the opposite. While I think that it is not desirable to have concrete mixed too dry, and equally undesirable to have it mixed too wet, yet if there is any question as to a little excess of water, or a little deficiency of water, we are generally a little bit safer in using a trifle more water than less, especially in cold and easily flooded localities.

Mr. Johnson. MR. JOHNSON.—There are members present who have not yet spoken, whose views I know differ from mine. I am deeply appreciative of the courteous motive which causes them to withhold remarks which might detract from the strength of my paper, but as general discussion is one of the most valuable parts of these meetings, I would ask that Mr. Bates and Mr. Spackman favor us with their opposing viewpoints.

Mr. Spackman. MR. H. S. SPACKMAN.—I disagree with the author in two of his conclusions, namely:

Mr. Spackma

First, I do not believe that the spots of unhydrated cement shown on the slides are due to the grouping of a number of very fine particles through surface tension. I believe these spots are due to the coarser particles in the cement, and that the globular or honeycombed appearance is simply the characteristic structure of the unhydrated cement clinker. When produced in the rotary kiln, the clinker is built up by agglomerations of numerous particles having a more or less globular shape. This is plainly visible to the naked eye on examining a piece of clinker; and the same globular structure in finer state of sub-division will be found to persist through the mass when it is examined under the microscope.

Second, I do not believe that the cracks shown on the photographs, radiating from the various voids, have been caused by crystallization of salts in these voids. It is my opinion that these are shrinkage cracks, forming lines of incipient weakness, whether they are filled with dikes of intruded material or not, and that they have been present in the concrete from the very beginning of the set; although they may have been opened up through the expansive force of the salts crystallizing in them.

I wish also to emphasize the author's recommendation of leaner mortars. The percentage of cementing material in all sandstone is very small, and the grains are close together. The close juxtaposition of the sand grains is also noticeable where lime is used in making concretes. We had occasion some years ago to make slides of a number of mortars taken from concrete from Roman ruins in Italy. In no case was there any wide space between the grains of sand, the ratio of lime to sand in the mortar being, as nearly as we could calculate, 1 to 6. This is also shown in slides made from sand-lime brick. In these the ratio of cementing material to sand is about 1 to 16 by weight, or 1 to 8 by volume.

The floating apart of the sand by the cement, shown by the author's photographs, suggests to me that our method of void determination is inaccurate. In the majority of cases the voids are determined on the dry sand. It seems to me the proper way would be to thoroughly puddle and settle the sand with an excess of water before determining the voids, as this will be the condition of the sand after the concrete has been placed.

Mr. Bates. MR. P. H. BATES.—I do not agree with Mr. Johnson on several points, but before discussing these I wish to refer to Mr. Larned's remarks. He spoke very strongly against wet concrete. I am in favor, on the other hand, of incorporating still more water into concrete. If the very wet mixes which are now used were allowed to remain in the mixer for a longer period by being kept in motion, it would be found that they would become very dry; in fact, so dry that to remove them from the mixer would require the addition of more water. If instead of removing them after the addition of this water, they were kept still longer in the mixer they would again become dry. On the addition of more water they would again become plastic. This could be repeated several times. In fact, at the Bureau of Standard's laboratory in Pittsburgh, we have mixed concrete for 8 hours in a cubic mixer. This was about 3 hours past the time of the final set of the cement. The result of this long mixing was a very plastic concrete. It was impossible to trowel the mortar, the concrete tending to string out after the trowel; and furthermore it was very good concrete, attaining very desirable strengths. This is nothing new. It was shown a number of years ago by Mr. Howard at the Watertown Arsenal.

Another feature about cement, which is possibly not so well known, is the ability of the set cement to take up further water. By the set cement, I mean that part of the cement which is hydrated. It is generally agreed that this latter material is largely colloidal. Colloids have the property of taking up water in indefinite and almost unlimited amounts; consequently the colloidal particles formed at the early periods take up water for a long time afterwards. A neat briquette examined at the early periods is sandy; examined at later periods it is found to be dense and vitreous looking. This change is due almost entirely to the absorption of water by the material which has set at the early period.

Returning to Mr. Johnson's paper, I cannot agree that fissures are produced by crystallization. I think on the other hand that crystallization is subsequent to the production of fissures. I do not agree with Mr. Johnson, furthermore, that agglomerates which he refers to as being composed of very fine particles of cement, are such. I think on the other hand that

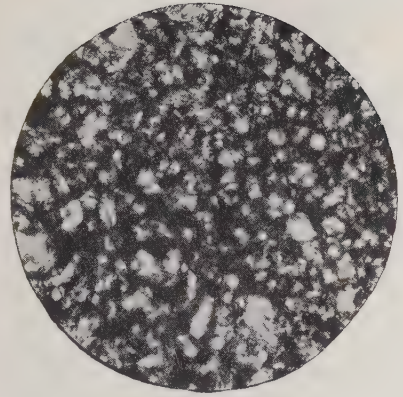


Fig. 1 (× 125).

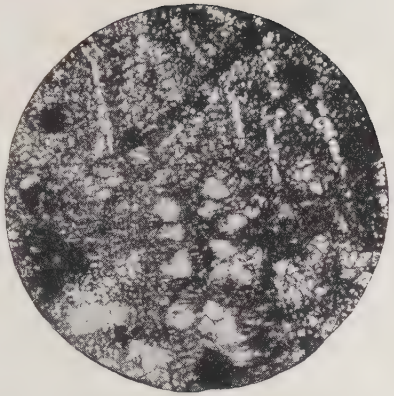


Fig. 2 (× 60).

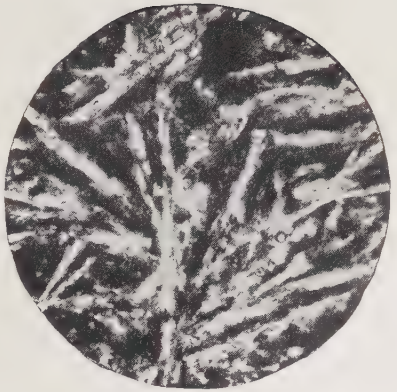


Fig. 3 (× 125).

Three microphotographs showing the structure of Portland cement clinker, when examined by transmitted light in thin sections with crossed nicols. Fig. 1 shows a structure similar to that exhibited by Mr. Johnson's "agglomerates." The dark material is the high double-refracting material containing iron. The white material consists of the compounds not colored by iron. Under certain conditions of composition and methods of manufacture, the granular form exhibited in Fig. 1 tends toward the needle formation which is shown to some extent in Fig. 2. Fig. 3 shows the needle formation in very large amounts. Average normal clinker is of the appearance shown by Fig. 1. Fig 3 shows clinker of unusual occurrence.

Mr. Bates. they are individual particles. I have a few slides (Figs. 1 to 3) that I will show which I think will help to make this clear. These are thin sections of clinker examined by transmitted light. In their preparation water had never touched them. Consequently, they are clinker as produced in the kiln. You will note in these slides the similarity between the structure of the clinker as made and the particles as shown by Mr. Johnson. You will note furthermore that the greater part of the constitutents of the clinker have this structure, both the brown constituent, which is the major silicate present in Portland cement, and the lighter crystallin material, which is a minor silicate.

Mr. Johnson. MR. JOHNSON.—May I say one word in closure? I asked Mr. Spackman and Mr. Bates to speak because I knew how well qualified they were, and because I was aware of the points they were about to bring out. If my assumption that these are agglomerations of cement particles is incorrect, I am very regretful, because I can then see no present way by which we can increase hydration. If they are agglomerations, I see a possibility for increasing the hydration and the strength of concrete by very simple means, and I sincerely hope that the measurements I have made may not prove in the long run to be erroneous.

Mr. Rinald. MR. C. D. RINALD (*by letter*).—Mr. Johnson has given a very lucid explanation of the action of water as one of the chief and probably least appreciated agents in the formation of concrete. His remarks on that point and those made in the discussion by two other gentlemen, the one speaking on thorough hydration and the other on addition of a sodium soap, bring to mind a method used with apparent success a number of years ago by the late Mr. Kindl. In a paper on "Waterproofing,"[1] after describing various methods of waterproofing cement, I suggested that good results ought to be obtained by imparting to the water colloidal qualities with a view of obtaining a better and more intimate union of the solid components of the mixture. My advice, based on Candlot's experiments, was to add about half a pound of calcium chloride to every bucket ($2\frac{1}{2}$ gallons) of water, and to use this solution just like plain water in mixing the concrete. Some two or three years later Mr. Kindl told

[1] *The Technologist*, Vol. 12, No. 7 (July, 1907).

me that he had tried out this procedure in the basement of a large office building and had since recommended it elsewhere. Mr. Rinald.

Let me add that it is important that for each job the amount of calcium chloride required should be determined by the man in charge. It will as a rule vary between one and two per cent. When the right proportion is reached, the mixture offers less resistance to troweling or tamping and packs closer than it does if mixed with plain water. An excess of calcium chloride accelerates the initial set and must be avoided as favoring the formation of cavities within the mass.

MR. WIG (*by letter*).—Mr. Johnson makes a valuable contribution to those investigating the characteristics and behavior of Portland cement concretes under various conditions, by suggesting the more widespread use of the microscope. Mr. Wig.

He makes, however, certain claims of discovery of the causes of porosity, of "secondary disintegrating actions," of unhydrated cement, and the cause of unhydration as due to surface tension of water which the writer desires to discuss.

From general knowledge of the large relative absorption and porosity of concretes, ranging from 2 to 20 per cent, as compared with natural stones, ranging from 0.01 to 5.0 per cent, engineers have long recognized that concrete was relatively a very porous material, filled with interspace, and that the individual particles were widely separated. Generally, natural stones having an absorption comparable with that of concrete have a comparable strength. Mr. Johnson has not suggested any new method to obtain greater compactness, but reiterates the generally accepted good recommended practice of careful mixing, the use of good materials, with the proper quantity of water, thorough compacting and careful curing.

He refers to certain markings observed on cement specimens, as "shear planes," "crystallin inclusions in shear planes," "dikes," "disruptive crystallin dikes," etc. These he observed on microscopic specimens with magnification ranging from 40 to 130 diameters and offers them in explanation of the development of cracks and fissures found in certain sidewalks, retaining walls, and dams. Granting that his contentions are correct, that "shear planes" do exist on such a microscopic scale, and necessarily with much frequency if they occur at all in a struc-

Mr. Wig. ture, it would seem that since they must result from some abnormal reactions of the cement particles of the matrix, they should cause complete disintegration and crumbling of the concrete rather than the formation of large fissures and cracks with hard sound edges bounding these cracks.

Because of the fineness of cement these abnormally behaving particles, if such exist, must be thoroughly scattered through the concrete. It is the belief of the writer that at least some of Mr. Johnson's so-called "shear planes" are incipient or pronounced cracks caused by strain, or existing since the original fabrication of the concrete. Examination of the interior of normal concrete often reveals fissures emanating from large particles of aggregate or groups of particles, apparently caused from settlement and readjustment in the mass during the setting period as the excess water works to the surface.

The "crystallin inclusions" referred to by Mr. Johnson are common to all porous or fissured concrete structures, subject to the flow of seepage water or to the absorption of saline waters. An analysis of these "crystallin inclusions" in the one case usually shows a lime carbonate, the lime having been dissolved from the cement by percolating waters brought to the surface of the structure and carbonated upon exposure to the air. Where the concrete is exposed to saline waters, an analysis of the salts in the fissure usually shows it to be similar to the salts of the water combined with lime or other elements taken from the cement. If these "crystallin inclusions" were the cause of the fissures, as Mr. Johnson seems to infer as resulting from peculiar "secondary disintegrating actions" of the cement, they would not be found only in poorly fabricated concretes, but also in well-fabricated concretes.

The statement that surface tension of the water is largely a contributing cause of unhydration of cement does not appear to be well founded. In practice the water is not poured upon the cement and absorbed as Mr. Johnson illustrates, but the cement particles are rubbed upon one another and ground and rubbed together between the sand and stone particles. Observation of gaged cement from a normally mixed batch of concrete shows no appreciable difference in appearance under the microscope whether mixed with a water-alcohol solution of low

surface tension or plain water, and tests of concrete mixed with such solutions gave the following results, which would tend to disprove the theory that surface tension is a factor: Mr. Wig.

One part Portland cement, two parts sand, four parts gravel, mixed quaking consistency: Specimens: 8 by 16-in. cylinders; age 30 days.

SOLUTION USED IN MIXING.	COMPRESSIVE STRENGTH, LB. PER SQ. IN.
Water	1660
5-per-cent annyl alcohol water solution	885

Mr. Johnson has shown that cement remains unhydrated in concretes even after a period of years and in concretes exposed to moisture continuously, but the writer does not find any data in the paper to warrant the conclusion that the presence of this unhydrated cement causes or contributes to disintegration. Furthermore, the results presented do not warrant the statement that microscopic examination is highly advantageous in determining the causes of failure of concrete, for as yet it has not accomplished this result, although we all hope it may ultimately do so.

MR. JOHNSON (*Author's closure by letter*).—That the preliminary results presented by this comparatively limited microscopic investigation of concretes has provoked so much interest and discussion by those best qualified to judge, is a very real indication of its value and possibilities. It is highly probable, as has been explicitly stated, that many of the conclusions arrived at will be subject to very radical revision and some possibly proven entirely erroneous. It seems probable to the author that Mr. Spackman's views as to the minute cracks first originating in shrinkage, and growing by the expansion of crystallin material, has more to recommend it than the view first held by the author that the fissures were formed solely by expansive crystallization in pores. Mr. Johnson.

In spite of the apparent identity between Mr. Bates' photographs and those shown by the author, and taken to be groupings of unhydrated cement particles, the author ventures to hope that there may be a possibility of his interpretation of these appearances being correct. If this is so, and if, by some means, dispersion can be effected, a substantial improvement in the quality of concrete should result.

Mr. Johnson.

Mr. Wig apparently holds that this study of concretes by means of the microscope has contributed nothing to our knowledge; and that it has resulted only in iteration and reiteration of long-known principles as to fabrication. It is regretably true, however, that although the principles may, as Mr. Wig says, have been well-known, they have yet been persistently and commonly disregarded in every-day work, in spite of previous warnings. If this microscopic method of examination shall have served in any degree to bring home the truth of these concrete axioms and to have brought about their application in to-day's work, it will have rendered a beneficial service to the industry, even though it should have fallen short of the mark in all other respects.

With respect to the value of lowered surface tension, Mr. Wig is too thorough a scientific investigator to reject any suggestion for improved procedure on the inconclusive evidence of a single adverse test, or even of a single series of adverse tests, particularly in view of the wide variation in results in even the most conscientious investigations, due to that obscure and elusive variable known as the "personal equation." Many of the author's test results show exceedingly wide variations, according as more or less of any given deflocculator is added; and much experiment is needed to determine the proper percentage and the best material to be used. A comprehensive seres of tests of this nature by the Bureau of Standards should be of value.

Mr. Larned is to be commended for his expressions in regard to the use of over-wet concrete and as to the care necessary in the selection and proportioning of the materials of concrete. Regardless of how well known the principles of correct fabrication may be, the fact remains that essentially all are either persistently and flagrantly violated, or else simply neglected, in order that larger profits may be realized. Nor does this apply alone to the constructor. The demand of the buyer for a low price and the keenness of competitive bidding, coupled with an unfortunately fostered idea that any mixture of cement, stone, sand and water is imperishable, have brought about these conditions. The next ten years will see a radical change in the procedure of making concrete.

The author has found special pleasure in Mr. Thompson's

expressions with regard to the need of putting microscopic investigation on a scientific basis. The primary object in making public the results of the writer's researches was the stimulation of general interest so that a more accurate and careful research in regard to concrete and the materials of concrete might be brought about. There are few who have conducted microscopic examinations of concrete in the past, but it is hoped that it will become a very general method of examination. Other methods, such as chemical methods, play their part, but they have not proved sufficient in the past to explain the ills of concrete; and as their aid, it is believed, microscopic examination will speedily become a widely recognized reliance. Mr. Johnson.

AN INVESTIGATION OF THE STRENGTH AND ELASTIC PROPERTIES OF CONCRETE-FILLED PIPE COLUMNS.

By F. W. Swain and A. F. Holmes.

SUMMARY.

Tests made on concrete-filled pipe columns, filled by the jolting process described in this paper, showed the columns to have a definite elastic limit and up to this load to be perfectly elastic, the steel pipe preventing permanent set from taking place in the enclosed concrete.

The concrete blocks made by this process were also found to have an elastic limit, although permanent set was found at the lowest loads.

The column loads at the elastic limit were distributed between the concrete and the steel, and it was found that the former checked almost exactly the stress at the elastic limit obtained by testing the blocks of plain concrete.

The results of these tests would indicate that a load of 25 per cent of the ultimate could be taken as a safe working load, which conforms very closely with the present practice.

Similar tests on larger columns are needed to complete this series and especially tests on columns containing a steel core.

AN INVESTIGATION OF THE STRENGTH AND ELASTIC PROPERTIES OF CONCRETE-FILLED PIPE COLUMNS.

By F. W. Swain and A. F. Holmes.

The object of the series of tests described in this paper was to determine whether a column, made by filling a steel pipe with concrete, was perfectly elastic under the lower loads, whether it had a definite elastic limit, and if such proved to be the case, to determine the stress in the concrete and in the steel at this load.

The columns were furnished by the Milford Iron Foundry of Milford, Mass., and were tested under the direction of their engineer, Mr. Swain. With the exception of columns Nos. 32 and 35, which were tested at the Watertown Arsenal, all tests were made on the 300,000-lb. Emery testing machine at the Massachusetts Institute of Technology between December 12 and 29, 1914; that is, at ages varying from 65 to 82 days. The columns ranged in size from a 4-in. light-weight boiler tube to a $6\frac{5}{8}$-in. standard-weight steam pipe, and were all filled on October 8, 1914, using a 1 : 2 : 3 mixture of concrete with $\frac{1}{2}$-in. screened, crushed stone, 8 by 8 by 24-in. test blocks of the concrete being cast at the same time and in the same way.

The methods employed in the making of these test columns were exactly the same as those used in the manufacture of all Milford Concrete-Filled Columns. The pipes were placed on end on the flat table of a specially constructed machine, and were then filled with machine-mixed concrete from an over-head hopper. Air was admitted to a cylinder under the table of this machine, lifting the machine some two inches. A release of the air allowed the table with its load to drop freely, and with a considerable shock, upon the heavy base of the machine. This jarring process was continued at the rate of about 150 shocks per minute, concrete being added at intervals, until there was no room for more.

It may be stated that an application for a patent on this type of column has been rejected.

After the concrete had become somewhat seasoned the ends of the columns were trued in the following manner: Each column

was strapped in V-blocks on the horizontal table of a cup-wheel grinder and the wheel was brought up against the end of the column, then carried across the end, at right angles to the axis of the column. After each trip across, the grinder was set a little ahead before starting the return cut. This gave a perfectly square cut across the full area of the pipe and concrete. The column was then turned end for end and the grinding repeated. In order to obtain uniform and intelligible results it was of course absolutely necessary that both ends of the column be square and parallel.

The average metal area in the cross-section was determined from the weight of the pipe before it was filled, and this value was used in making all computations.

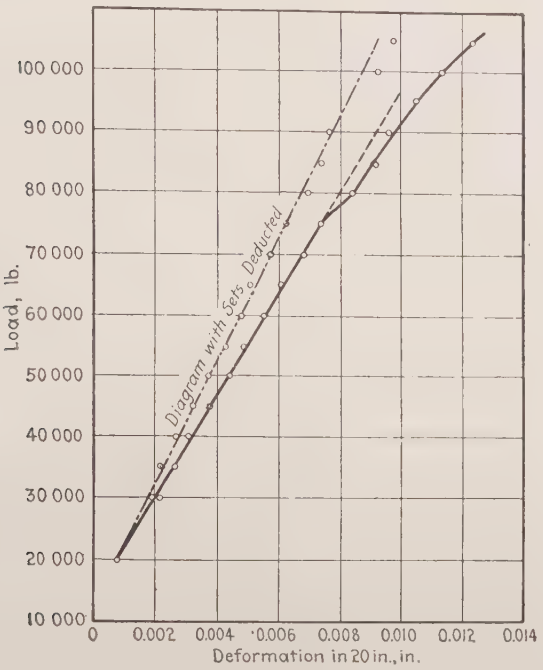
TABLE I.—CONCRETE BLOCK SPECIMENS—MIXTURE 1 : 2 : 3.

Specimen.	Size, in.	Weight, lb.	Ultimate Load, lb.	Ultimate Strength, lb. per sq. in.	Elastic Limit, lb. per sq. in.
A	8 by 8 by 24	135.0	167 700	2620[a]	75 000
B	" " "	136.5	260 900	4080	.....
C	" " "	138.0	259 200	4050	.....
D	" " "	135.5	260 300	4070	.....
E	" " "	136.5	256 800	4020	.....
				Aver. = 4050	

[a] The low ultimate strength of specimen A was due to the repeated application of the load and this value was therefore omitted from the average.

The results of the tests on the concrete block specimens, five in number, are given in Table I. Deformations in a gage length of 20 in. were measured on specimen A to determine the modulus of elasticity of the concrete; the readings and stress-deformation diagram are given in Fig. 1.

The results of the tests on the columns are given in Table II. On columns Nos. 3, 9, 14, 15, 20, 25 and 33, deformations in a gage length of 32 in. were measured and the load at which the permanent set became a measurable quantity was determined (this load is referred to as the "elastic limit" hereafter). The readings and stress-deformation diagrams for these seven columns are given in Figs. 2 to 8, inclusive. Reference to these readings



Load, lb.	Average Reading, in.	Difference, in.	Sum, in.	Load, lb.	Average Reading, in.	Difference, in.	Sum, in.
10 000	0.3024	0.0000	0.0000	10 000	0.3032	0.0008	0.0008
20 000	0.3032	0.0008	0.0008	65 000	0.3085	0.0053	0.0061
10 000	0.3024	0.0000	0.0000	10 000	0.3034	0.0010	0.0010
30 000	0.3043	0.0019	0.0019	70 000	0.3092	0.0058	0.0068
10 000	0.3026	0.0002	0.0002	10 000	0.3034	0.0010	0.0010
30 000	0.3045	0.0019	0.0021	75 000	0.3098	0.0064	0.0074
35 000	0.3050	0.0005	0.0026	10 000	0.3035	0.0011	0.0011
10 000	0.3028	0.0004	0.0004	80 000	0.3108	0.0073	0.0084
40 000	0.3055	0.0027	0.0031	10 000	0.3038	0.0014	0.0014
10 000	0.3028	0.0004	0.0004	85 000	0.3115	0.0077	0.0091
45 000	0.3062	0.0034	0.0038	10 000	0.3041	0.0017	0.0017
10 000	0.3029	0.0005	0.0005	90 000	0.3120	0.0079	0.0096
50 000	0.3068	0.0039	0.0044	10 000	0.3043	0.0019	0.0019
10 000	0.3030	0.0006	0.0006	95 000	0.3129	0.0086	0.0105
55 000	0.3073	0.0043	0.0049	100 000	0.3138	0.0009	0.0114
10 000	0.3030	0.0006	0.0006	10 000	0.3045	0.0021	0.0021
60 000	0.3080	0.0050	0.0056	105 000	0.3147	0.0102	0.0123
				10 000	0.3049	0.0025	0.0025

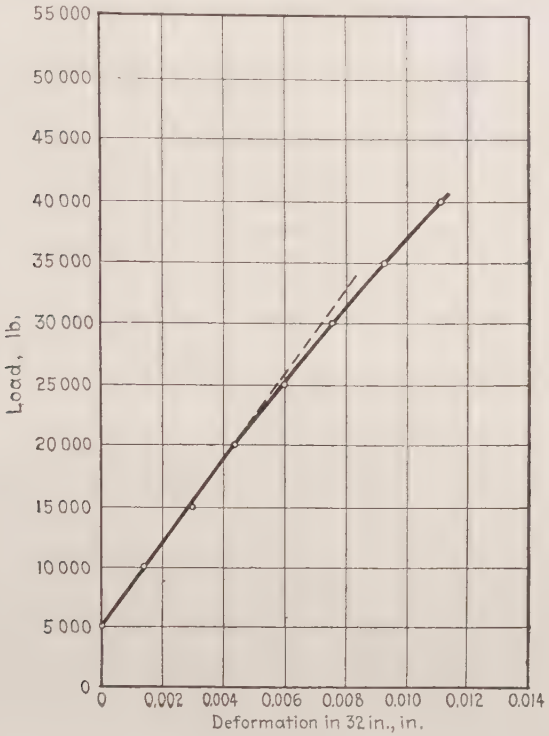
Dimensions = 8 by 8 by 24 in. Gage length = 20 in. Age = 76 days.
Ultimate load = 167,700 lb. Ultimate strength = 2620 lb. per sq. in.

FIG. 1.—CONCRETE SPECIMEN A

TABLE II.—RESULTS OF TESTS ON CONCRETE-FILLED PIPE COLUMNS.

Column No.	Outside Diameter, in., and Kind.	Nominal Thickness, in.	Length, ft.	Ratio of Length to Diameter.	Weight, lb.		Ultimate Load, lb.	Average of Three Tests, lb.	Elastic Limit, lb.	Elastic Limit / Ultimate Load.
					Unfilled.	Filled.				
1	4—Light	9/64	8	24.0	44.5	137	85 400	88 700	20 000 (No. 3)	0.227
2	4— "	5/32	8	24.0	47.0	139	92 600			
3	4— "	9/64	8	24.0	46.0	139	88 200			
4	4— "	9/64	10	30.0	55.0	169	67 900	74 900		
5	4— "	9/64	10	30.0	61.0	168	78 400			
6	4— "	9/64	10	30.0	57.0	165	78 300			
7	5— "	5/32	10	24.0	80.0	263	165 100	160 100	45 000 (No. 9)	0.269
8	5— "	11/64	10	24.0	82.0	258	148 000			
9	5— "	5/32	10	24.0	80.0	265	167 300			
10	5— "	5/32	14	33.6	115.5	370	144 800	143 100		
11	5— "	5/32	14	33.6	110.5	364	145 300			
12	5— "	5/32	14	33.6	105.5	360	139 300			
13	6— "	11/64	10	20.0	101.0	365	210 000	241 200	56 000 (No. 14)	0.214 } Aver. = 0.253
14	6— "	3/16	10	20.0	104.0	377	261 600		73 500 (No. 15)	0.292 }
15	6— "	3/16	10	20.0	105.0	377	251 900			
16	6— "	3/16	14	28.0	147.0	522	199 900	204 300		
17	6— "	11/64	14	28.0	145.0	520	243 700			
18	6— "	3/16	14	28.0	144.5	512	169 300			
19	4—Heavy	7/32	8	24.0	69.0	155	147 400	144 300	40 000 (No. 20)	0.277
20	4— "	15/64	8	24.0	72.5	159	144 700			
21	4— "	7/32	8	24.0	71.0	156	140 900			
22	4— "	1/4	10	30.0	90.0	194	130 900	128 700		
23	4— "	15/64	10	30.0	91.0	194	133 100			
24	4— "	15/64	10	30.0	92.0	198	122 200			
25	5— "	1/4	10	24.0	124.5	295	200 200	200 000	60 000 No. 25)	0.299
26	5— "	15/64	10	24.0	120.0	295	199 900			
27	5— "	15/64	10	24.0	121.0	291	199 900			
28	5— "	1/4	12	28.8	144.5	345	180 500	196 500		
29	5— "	1/4	12	28.8	151.0	357	199 900			
30	5— "	1/4	12	28.8	148.5	356	208 900			
31	6 5/8— "	9/32	12	21.7	222.0	592	Not tested	304 200	90 000 (No. 33)	0.295
32	6 5/8— "	17/64	12	21.7	215.0	592	303 300			
33	6 5/8— "	9/32	12	21.7	221.5	597	305 000[a]			
34	6 5/8— "	9/32	16	29.0	286.5	790	300 000 (not broken)			
35	6 5/8— "	9/32	16	29.0	304.0	767	274 900			
36	6 5/8— "	19/64	16	29.0	321.5	801	300 000 (not broken)			

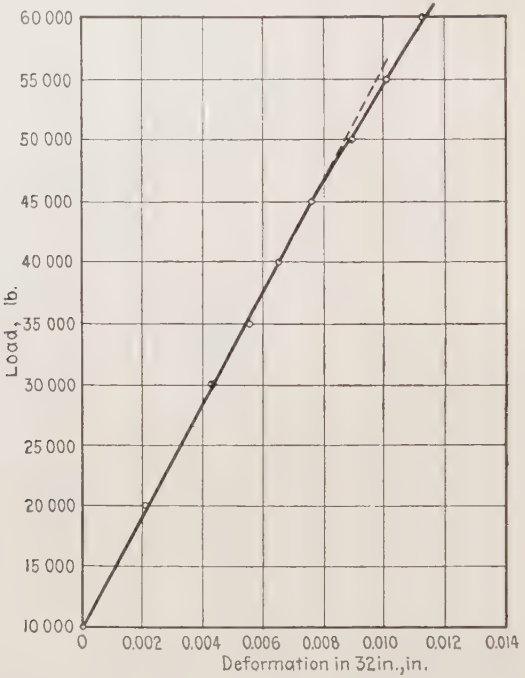
[a] Failed at a load slightly above rated capacity of machine and was assumed to be 305,000 lb.



Load, lb.	Reading, in.			Difference, in.	Sum, in.
	North.	South.	Average.		
5 000	0.0292	0.0296	0.0294	0.0000	0.0000
10 000	0.0293	0.0322	0.0308	0.0014	0.0014
15 000	0.0300	0.0347	0.0324	0.0016	0.0030
20 000	0.0308	0.0365	0.0337	0.0013	0.0043
25 000	0.0319	0.0388	0.0354	0.0017	0.0060
30 000	0.0333	0.0407	0.0370	0.0016	0.0076
5 000	0.0293	0.0298	0.0296	0.0002	0.0002
35 000	0.0347	0.0425	0.0386	0.0090	0.0092
5 000	0.0299	0.0300	0.0300	0.0006	0.0006
40 000	0.0364	0.0446	0.0405	0.0105	0.0111
5 000	0.0293	0.0301	0.0297	0.0003	0.0003

Diameter = 4 in. Length = 8 ft. Age = 81 days.
Gage length = 32 in. Ultimate load = 88,200 lb.

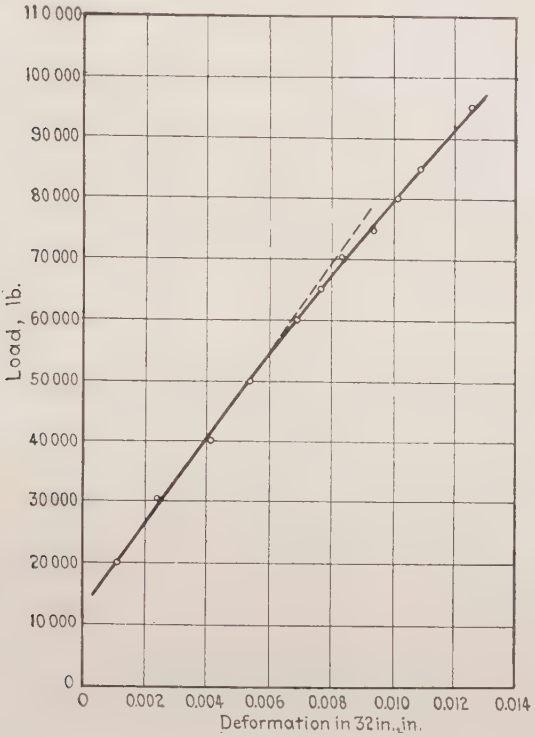
FIG. 2.—COLUMN NO. 3.



Load, lb.	Average Reading, in.	Difference, in.	Sum, in.
10 000	0.0471	0.0000	0.0000
20 000	0.0492	0.0021	0.0021
30 000	0.0514	0.0022	0.0043
35 000	0.0526	0.0012	0.0055
10 000	0.0471	0.0000	0.0000
40 000	0.0536	0.0065	0.0065
45 000	0.0547	0.0011	0.0076
50 000	0.0560	0.0013	0.0089
10 000	0.0473	0.0002	0.0002
55 000	0.0572	0.0099	0.0101
10 000	0.0474	0.0003	0.0003
60 000	0.0584	0.0110	0.0113
10 000	0.0474	0.0003	0.0003

Diameter = 5 in. Length = 10 ft. Age = 81 days.
Gage length = 32 in. Ultimate load = 167,300 lb.

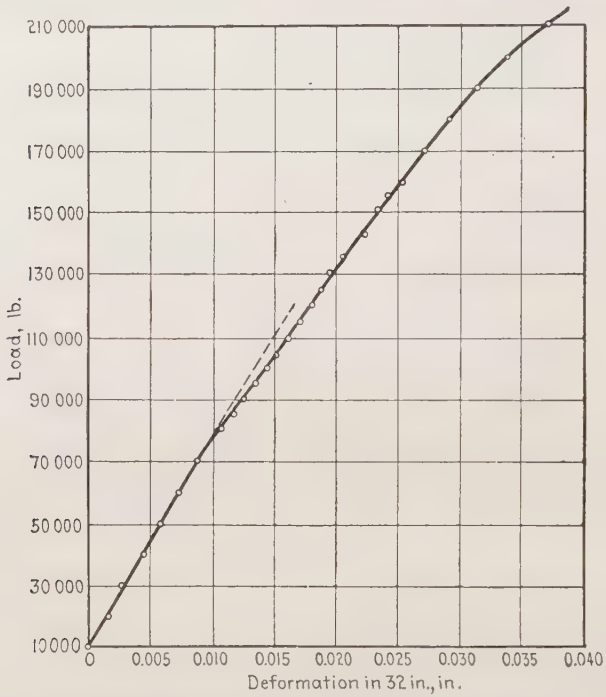
FIG. 3.—COLUMN NO. 9.



Load, lb.	Average Reading, in.	Difference, in.	Sum, in.	Load, lb.	Average Reading, in.	Difference, in.	Sum, in.
10 000	0.0098	0.0000	0.0000				
20 000	0.0110	0.0012	0.0012	10 000	0.0098	0.0000	0.0000
30 000	0.0123	0.0013	0.0025	75 000	0.0192	0.0094	0.0094
40 000	0.0139	0.0016	0.0041				
50 000	0.0152	0.0013	0.0054	10 000	0.0099	0.0001	0.0001
				80 000	0.0199	0.0100	0.0101
10 000	0.0098	0.0000	0.0000				
50 000	0.0152	0.0054	0.0054	10 000	0.0099	0.0001	0.0001
60 000	0.0167	0.0015	0.0069	85 000	0.0207	0.0108	0.0109
10 000	0.0098	0.0000	0.0000	10 000	0.0100	0.0002	0.0002
65 000	0.0175	0.0077	0.0077	95 000	0.0223	0.0123	0.0125
10 000	0.0098	0.0000	0.0000				
70 000	0.0182	0.0084	0.0084				

Diameter = 6 in. Length = 10 ft. Age = 71 days
Gage length = 32 in. Ultimate load = 261,600 lb.

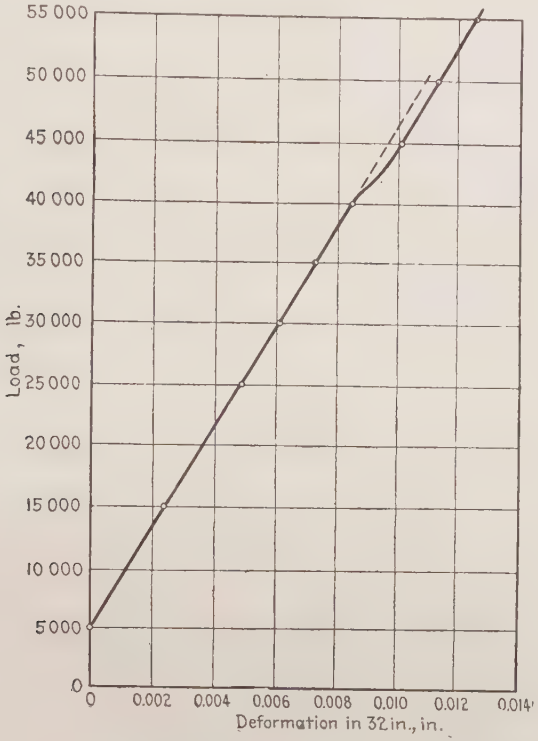
FIG. 4.—COLUMN NO. 14.



Load, lb.	Average Reading, in.	Difference, in.	Sum, in.	Load, lb.	Average Reading, in.	Difference, in.	Sum, in.	Load, lb.	Average Reading, in.	Difference, in.	Sum, in.
10 000	0.0779	0.0000	0.0000	10 000	0.0781	0.0002	0.0002	10 000	0.0790	0.0011	0.0011
20 000	0.0793	0.0014	0.0014	80 000	0.0886	0.0105	0.0107	120 000	0.0960	0.0170	0.0181
				85 000	0.0895	0.0009	0.0116				
10 000	0.0779	0.0000	0.0000					10 000	0.0792	0.0013	0.0013
20 000	0.0793	0.0014	0.0014	10 000	0.0782	0.0003	0.0003	125 000	0.0967	0.0175	0.0188
30 000	0.0808	0.0015	0.0029	85 000	0.0895	0.0113	0.0116				
				90 000	0.0903	0.0008	0.0124	10 000	0.0788	0.0009	0.0009
10 000	0.0779	0.0000	0.0000					130 000	0.0976	0.0188	0.0197
30 000	0.0807	0.0028	0.0028	10 000	0.0781	0.0002	0.0002				
40 000	0.0824	0.0017	0.0045	90 000	0.0903	0.0122	0.0124	10 000	0.0789	0.0010	0.0010
				95 000	0.0912	0.0009	0.0133	135 000	0.0986	0.0197	0.0207
10 000	0.0779	0.0000	0.0000					145 000	0.1003	0.0017	0.0224
40 000	0.0822	0.0043	0.0043	10 000	0.0784	0.0005	0.0005				
50 000	0.0838	0.0016	0.0059	95 000	0.0913	0.0129	0.0134	10 000	0.0792	0.0013	0.0013
				100 000	0.0922	0.0009	0.0143	150 000	0.1012	0.0220	0.0233
10 000	0.0779	0.0000	0.0000					155 000	0.1022	0.0010	0.0243
50 000	0.0839	0.0060	0.0060	10 000	0.0784	0.0005	0.0005	160 000	0.1033	0.0011	0.0254
60 000	0.0853	0.0014	0.0074	100 000	0.0922	0.0138	0.0143				
				105 000	0.0931	0.0009	0.0152	10 000	0.0795	0.0016	0.0016
10 000	0.0779	0.0000	0.0000					170 000	0.1051	0.0256	0.0272
60 000	0.0853	0.0074	0.0074	10 000	0.0786	0.0007	0.0007	180 000	0.1072	0.0021	0.0293
70 000	0.0868	0.0015	0.0089	110 000	0.0941	0.0155	0.0162	190 000	0.1094	0.0022	0.0315
								200 000	0.1118	0.0024	0.0339
10 000	0.0779	0.0000	0.0000					210 000	0.1150	0.0032	0.0371
70 000	0.0869	0.0090	0.0090	10 000	0.0788	0.0009	0.0009	220 000	0.1206	0.0056	0.0427
80 000	0.0885	0.0016	0.0106	115 000	0.0950	0.0162	0.0171				

Diameter = 6 in. Length = 10 ft. Gage length = 32 in. Ultimate load = 251,900 lb. Age = 65 days.

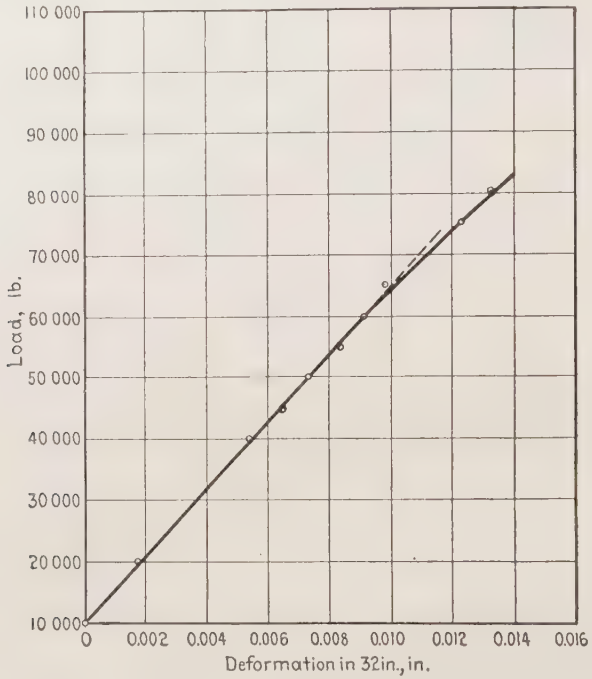
FIG. 5.—COLUMN NO. 15.



Load, lb.	Average Reading, in.	Difference, in.	Sum, in.
5 000	0.0253	0.0000	0.0000
15 000	0.0277	0.0024	0.0024
25 000	0.0302	0.0025	0.0049
30 000	0.0314	0.0012	0.0061
35 000	0.0326	0.0012	0.0073
5 000	0.0253	0.0000	0.0000
40 000	0.0338	0.0085	0.0085
45 000	0.0354	0.0016	0.0101
5 000	0.0255	0.0002	0.0002
50 000	0.0366	0.0111	0.0113
5 000	0.0256	0.0003	0.0003
55 000	0.0379	0.0123	0.0126
5 000	0.0256	0.0003	0.0003

Diameter = 4 in. Length = 8 ft. Age = 81 days.
Gage length = 32 in. Ultimate load = 144,700 lb.

FIG. 6.—COLUMN NO. 20.

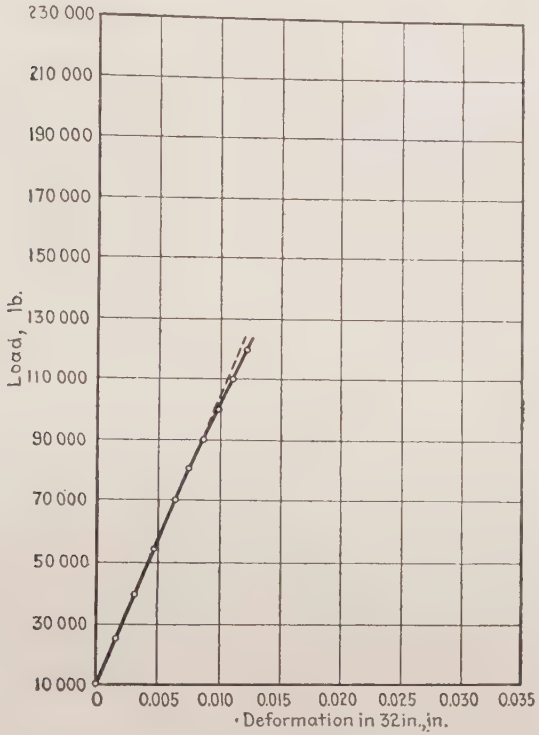


Load, lb.	Average Reading, in.	Difference, in.	Sum, in.	Load, lb.	Average Reading, in.	Difference, in.	Sum, in.
10 000	0.0456	0.0000	0.0000	10 000	0.0461	0.0000	0.0000
20 000	0.0473	0.0017	0.0017	10 000	0.0457	0.0000	0.0000[b]
40 000	0.0510	0.0037	0.0054	70 000	0.0570	0.0113	0.0113
45 000	0.0519	0.0009	0.0063				
				10 000	0.0457	0.0000	0.0000
10 000	0.0461	0.0000	0.0000[a]	75 000	0.0580	0.0123	0.0123
45 000	0.0526	0.0065	0.0065				
50 000	0.0534	0.0008	0.0073	10 000	0.0458	0.0001	0.0001
				80 000	0.0590	0.0132	0.0133
10 000	0.0461	0.0000	0.0000				
55 000	0.0544	0.0083	0.0083	10 000	0.0458	0.0001	0.0001
60 000	0.0552	0.0008	0.0091				
10 000	0.0461	0.0000	0.0000				
65 000	0.0559	0.0098	0.0098				
70 000	0.0570	0.0011	0.0109				

Diameter = 5 in. Length = 10 ft. Age = 81 days.
Gage length = 32 in. Ultimate load = 200,200 lb.

[a] Rods changed giving new zero.
[b] Micrometer readjusted.

FIG. 7.—COLUMN NO. 25.



Load, lb.	Average Reading, in.	Difference, in.	Sum in.
10 000	0.0969	0.0000	0.0000
25 000	0.0985	0.0016	0.0016
40 000	0.1001	0.0016	0.0032
10 000	0.0969	0.0000	0.0000
55 000	0.1017	0.0048	0.0048
70 000	0.1033	0.0016	0.0064
80 000	0.1044	0.0011	0.0075
10 000	0.0969	0.0000	0.0000
90 000	0.1055	0.0086	0.0086
100 000	0.1067	0.0012	0.0098
10 000	0.0970	0.0001	0.0001
110 000	0.1079	0.0109	0.0110
10 000	0.0971	0.0002	0.0002
120 000	0.1091	0.0120	0.0122
10 000	0.0971	0.0002	0.0002

Diameter = 6⅝ in. Length = 12 ft.
Gage length = 32 in. Age = 82 days.
Failed at a load slightly over capacity of machine (300,000 lb.)

FIG. 8.—COLUMN NO. 33.

and diagrams show that the filled columns were perfectly elastic when subjected to the lower loads, no permanent set taking place until after the elastic limit was reached. It will be noticed that with the exception of column No. 20 (Fig. 6), the stress-deformation diagram consists of two straight lines intersecting at the elastic limit. Column No. 15 (Fig. 5) was carried far enough to show the diagram leaving the second straight line in a smooth curve.

Reference to the stress-deformation diagram for concrete specimen A (Fig. 1) shows that while concrete made by this process gives a straight-line diagram and has a definite elastic limit, permanent set begins at once. It is evident, therefore, that in the filled column the steel tube prevents the permanent set from taking place in the concrete below the elastic limit, and the assumption was therefore made that the stress-deformation diagram for the concrete, restrained by the pipe, was the same as would be obtained for the unrestrained concrete with the sets deducted. On this assumption and the further assumption that the modulus of elasticity of the steel pipe was 30,000,000 lb. per sq. in., we have the following, using the deformations recorded in Fig. 1:

From diagram with sets deducted:

$$E_c = \text{Modulus of Elasticity of Concrete} = \frac{55{,}000 \times 20}{64 \times (0.0074 - 0.0011 - 0.0008)} =$$

3,125,000 lb. per sq. in.

From diagram with sets not deducted:

$$E_c = \text{Modulus of Elasticity of Concrete} = \frac{55{,}000 \times 20}{64 \times (0.0074 - 0.0008)} =$$

2,605,000 lb. per sq. in.

$$\frac{E_s}{E_c} = \frac{30{,}000{,}000}{3{,}125{,}000} = 9.6$$

$$\text{Stress in concrete at elastic limit} = \frac{P}{A} = \frac{75{,}000}{64} = 1170 \text{ lb. per sq. in.}$$

Using this ratio of $E_s/E_c = 9.6$, the proportion of the load carried by the concrete and by the steel was determined and it was found that the stress in the concrete at the elastic limit, except in the case of column No. 3, checked almost exactly with

the stress in the unrestrained concrete at its elastic limit. Therefore, it is evident that the elastic limit of the column was determined by the elastic limit of the enclosed concrete. The elastic limits of the columns are given in Table III, as well as the computations for column No. 25, which are typical.

An explanation of the low value obtained in the case of column No. 3 was found upon inspection of the deformation

TABLE III.—ELASTIC LIMIT OF CONCRETE-FILLED PIPE COLUMNS.

Specimen.		Area of Steel, sq. in.	Total Equivalent Area, sq. in.	Stress in Concrete at Elastic Limit, lb. per sq. in.	Stress in Steel at Elastic Limit, lb. per sq. in.
Concrete.	Column.				
A	..	....	64.0	1170	.....
....	3	1.69	27.10	740	7 100
....	9	2.35	39.85	1130	10 800
....	14	3.06	54.58	1030 } Av. = 1190	11 400
....	15	3.08	54.74	1350 }	
....	20	2.66	35.46	1130	10 800
....	25	3.66	51.10	1170	11 200
....	33	5.43	81.16	1110	10 700

CALCULATIONS FOR COLUMN NO. 25.

Weight unfilled = 124.5 lb.
Outside diameter = 5 in.
Length = 10 ft.
Load at elastic limit = 60,000 lb.

$$\text{Area of steel in cross-section} = \frac{124.5 \times 144}{490 \times 10} = 3.66 \text{ sq. in.}$$

Area of concrete in cross-section = 19.64 − 3.66 = 15.98 sq. in.
Concrete equivalent of steel = 3.66 × 9.6 = 35.12 sq. in.
Total equivalent area = 15.98 + 35.12 = 51.10 sq. in.

$$\text{Stress in concrete at elastic limit} = \frac{60{,}000}{51.1} = 1170 \text{ lb. per sq. in.}$$

readings, which in this instance are given in full (see Fig. 2). It will be noticed that the compression was confined almost entirely to one side, but whether this eccentric loading was due to a defective pipe or improper bearing is not known.

An investigation of the ratio in each case of the elastic limit to the ultimate load (see Table II) would indicate that a load of at least 25 per cent of the ultimate would be a safe working load, and that an even higher value might be used in certain cases.

As a result of these tests the column formulas given in Fig. 9 were devised to take into account the stiffness of the column,

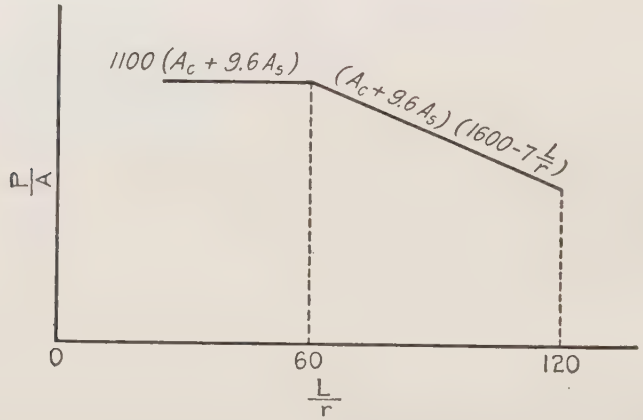


FIG. 9.—Formulas for Concrete-Filled Pipe Columns.

A_c = Area of concrete in cross-section.
A_s = Area of steel in cross-section.
E_c = Modulus of elasticity of concrete (sets deducted).
E_s = Modulus of elasticity of steel.
E_s/E_c = 9.6 (for 1 : 2 : 3 concrete used in these experiments).
L = Length of column.
r = Radius of gyration (solid section).
P/A = Safe working load (lb. per. sq. in.).

and they were used in compiling a table of safe working loads to which concrete-filled columns of this type could be subjected, when the values of L/r varied between 30 and 120. The formulas as given apply only to columns made of the same 1 : 2 : 3 mix of concrete as was used in filling the columns tested, but the constants could easily be modified to adapt them to any other mix.

THE LEGAL INTERPRETATION OF THE WORD "VITRIFIED" AS APPLIED TO CERAMIC PRODUCTS.

By Edward Orton, Jr.

SUMMARY.

The word "vitrified" is employed widely by engineers, architects, contractors, and others in describing the physical status of the following ceramic products, namely, paving bricks, sewer pipe, roofing tiles, drain tiles, encaustic flooring tiles, quarry tiles, face bricks, stoneware pottery, earthenware pottery, table porcelain, insulator porcelain, and others. In applying it to these different products, the word by implication and general usage means very different things in different cases. The reasons for this are fundamental, depending (1) upon the fact that vitrification is a process towards or through which all clay products pass in burning with markedly different results in different clays, and (2) upon the fact that different clay products require widely different degrees of vitrification to fit them for their respective purposes.

For the protection of clay-product manufacturers and dealers, who often suffer severely by the arbitrary enforcement of literal interpretations of the word, where trade usage did not contemplate such an interpretation at all, a definition of the word "vitrified" to apply to all cases where the specifications in question contain no definition of the term, has been suggested as follows:

"Where the word "vitrified" is used in a contract or agreement in describing the properties of a clay product, and is not further defined in the contract, it shall mean that the product shall have attained in the firing process such a degree of com-

pactness that its mean absorption does not exceed three per cent of its own initial weight of pure water, when subjected to complete immersion in cold water for forty-eight hours, or boiling water for three hours. Not less than three tests shall be made in determining the mean absorption, and no single test may exceed the mean by more than 0.5 per cent. Neither the possession of the visual properties or appearance of a glass, nor any definite degree of physical strength, hardness, color, or specific gravity shall be implied as an attribute of vitrification."

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The clay products of this country, exceeding the gross valuation of $180,000,000 annually, are as yet being marketed in a very immature and unscientific way. The larger part are marketed without any formal specifications, as is the case with the bulk of the common brick used. A considerable amount is also marketed under specifications couched in such general and undefined terms that they amount to little except as declarations of good faith between buyer and seller. In some other instances, general specifications are signed up which are usually treated as mere formalities, not to be taken literally, but which are nevertheless capable of making endless trouble and loss if a change of feeling causes them to be overhauled and applied with rigidity. It is difficult to say which form of laxity has cost the people more, that which uses undefined terms, or that which uses sharply defined terms on the understanding that they will not be enforced.

Clay products may be grouped into two great divisions, namely, Vitrified and Porous. This division extends into practically all branches of clay manufacture, whether utensils or construction materials. Such a distinction, for instance, may be drawn between common brick, fire brick, insulating bricks, hollow bricks, etc., as one group, and paving brick, and the better grade of face bricks in another; again, in quarry tiles, floor tiles, and hard roofing tiles as against wall tiles and soft roofing tiles; and again, in sewer pipes, chemical pipes and electric conduits as against drain tiles, building tiles and fireproofing. Similar comparisons could be made equally well in the pottery group.

The vitrification of the one group is its main title to excellence, while the porosity of the other is essential to the fulfilment

of its function. And yet, with such obvious and direct importance attached to this word "vitrified" no clear and generally accepted definition is in use, and the word is being used in diverse senses to the disadvantage of the whole public. No single step of more importance toward the standardization of clay products can be taken than the definition of this term.

The first great stumbling block in clearing up this unfortunate situation is the fact that there are two allied, but nevertheless clearly distinct, points of view in regard to the use of the term. These may be characterized as the chemical and the physical conceptions.

THE CHEMICAL MEANING OF VITRIFICATION.

Vitrification is understood among clayworkers to be the chemical process by which a clay is hardened by heat. It is essentially a process of silicate formation. A clay may be burnt at temperatures below 900° C. without any appreciable silicate formation taking place, in which case, there is virtually no increase in hardness or strength over the dried condition. At 900° C., or above, the granular structure begins to yield to the cementing action of silicates formed by combinations of the different minerals present. This cementing material at first is formed in small quantities, and if the burn is stopped at this stage, a weak, friable and porous product is formed. At higher temperatures it may be abundant enough to fill the voids and to thoroughly bind the unfused matter together. This produces a strong, tough product. In some clays, the whole mass of the mineral material may tend to combine at about the same time, forming altogether too much silicate matter to have the necessary stability of form. Such clays fuse or deform suddenly. No two clays show exactly the same behavior in respect to their silicate formation or vitrification.

The principal factors causing these variations in the process are as follows:

1. The mineral mixture, natural or artificial, of which the clay body consists.
2. The fineness of grain and homogeneity of the mineral mixture before heating.

3. The rate of heating, or the use of the time factor in bringing the minerals into combination with each other.

4. The temperature to which the heating is carried.

5. The chemical condition of the kiln atmosphere.

A discussion of the operation of these factors is not necessary to the present purpose of this paper. Suffice it to say that any one of the five factors is able to profoundly influence the duration of the process and the physical character of the product, and jointly they sufficiently explain why clays exhibit such individuality in their properties. Nevertheless, the nature of the process through which they pass is the same for all.

The beginnings of vitrification are in the formation of small foci or centers of fusion, in which a single fusible mineral grain, or two interacting grains form a minute quantity of fused mineral. This acts as a solvent or flux to neighboring grains. The material formed in these early foci may not be uniform in nature, but as the amount of material brought into solution gets larger, it becomes more uniform in type. As the process goes on, the amount of fused silicate solvent increases more and more, until after a time, the resistant framework formed by the solid grains is cut in pieces here and there by the inroads of the solvent, and the mass begins to gently sink out of shape. It has reached its deformation temperature and if the heat treatment is still kept up, the mass will ultimately pass from viscous solid to a real fluid.

The Differentiation of Vitrification and Fusion.—The key to the dividing line between these two stages is the physical state. In vitrification, the status of the body must remain that of a solid. Fluids are being formed within it during this process and the whole mass is approaching fusion, but we only recognize that fusion is reached when the mass will no longer support its own weight without deformation, and begins to obey the laws of a fluid.

The use of the terms "incipient vitrification," "complete vitrification," "viscous vitrification," etc., serves to convey the idea of progressive stages of a process tending towards fusion, but no definitely identifiable boundaries between these stages can be set, and hence they have little real value.

According to the chemical conception, vitrification is not

conditioned by the quantitative possession of any single physical property or group of properties, but is a process, or better, a stage in a process.

It will be apparent, I think, that between chemists or clayworkers, it is proper to speak of a clay product as completely vitrified if it has undergone its hardening process so far as its mineral composition enables it to go. This may be a long ways from dense, or non-porous, but the criterion in this use of the word is not how hard or dense it may be, but how far its chemical transformation has progressed. If any additional heat softens it and puts it over the line and into the group of fused bodies, then it is at its stage of complete vitrification, regardless of its physical structure at that stage.

In this sense clayworkers allude to the vitrification period (of the burning process); to a broad or narrow vitrification range (meaning the temperature interval between the beginning of hardening and the loss of the solid state); to its vitrification rate or speed, etc.

The Physical Conception of Vitrification.

The physical idea of vitrification deals with the possession of certain properties, without any regard as to how these properties have been developed, or whether they are as fully developed as they may be by variation of the vitrifying treatment, etc. This is the usual point of view of the engineer, contractor, and dealer. They are not at all concerned with or interested in the difficulties that the clayworker undergoes in the process, but devote their whole attention to determining whether he has brought his product to a certain state which they call "vitrified."

This view of the meaning of the word is also supported by its history, or derivation. In a previous article by the writer[1] upon this subject it was shown that from its Latin derivation, the verb "vitrify" means "to make glassy," and that all dictionaries, encyclopedias and books of reference preserve this signification unimpaired after two thousand or more years. This theoretical meaning is, however, subject to attack, (1) on the ground that it is not now and has not for a long period of time

[1] "The Legal Definition of Vitrification," *Transactions*, Am. Ceramic Soc., Vol. XVI, pp. 497–514 (1914).

been used at all in the glass industry—indeed its use is wholly foreign to that industry; and (2) that in the clay industry, where it is used largely, it does not and has not meant literally "to make glassy." Claywares burnt to a condition where visible glassiness exists are the very rare exception, and are usually defective from over-fire.

From this, it appears that the interests of the clay-product manufacturer are in danger from this old historic meaning of the word. Let a case arise where the law is invoked to settle a controversy as to whether a clayware is "vitrified" or not, without any other definition in the contract, and the lawyer will at once fall back on this idea of glassiness; and failing to find any glassiness in the product will make capital out of that fact and will usually win his case. It is absolutely necessary to shatter this old idea of glassiness as a condition of vitrification, for the protection of the clayworker, who cannot safely make his ware glassy if he wants to, and the clay-product user, who would suffer if glassy ware were actually furnished.

In this physical concept of vitrification, it is evident that there must be some quantitative measurement of the properties by which the matter can be decided. One man's opinion is as good as another's in a case which does not admit of measurement, and hence no decision is possible in contested cases, unless the properties of vitrification are defined and measured.

There are quite a number of such properties which may be used but only six need be considered, namely, color, texture, hardness, strength, specific gravity, and compactness or freedom from voids.

Color.—Changes in color have no necessary relation to silicate formation. Silicates of every color imaginable can be produced. Colors are therefore not direct indices to vitrification and can only be used as a guide in so far as their variations are found to be serial and regular, and correlatable with the other properties of vitrification. As between different clays, no sharp comparisons can be drawn. Between different stages of vitrification of a single clay, the color changes are commonly pretty regular and dependable. Between red-burning clays as a group, the variety and sequence of the color changes are fairly similar. The buff-burning clays exhibit a similar degree of comparability.

Furthermore, the measurement of color, *per se*, is not easy, and requires a skilled physicist, with abundant equipment.

The color index to vitrification will therefore probably remain as a crude and very imperfect guide. It may offer a very convenient visual test for one observer dealing with one clay, but it can never become of general application between different clays and different observers.

Texture.—The texture of a natural mineral or fused silicate may vary from a coarsely granular, or crystalline mass, through every degree of fineness, till an apparently structureless homogeneous glass is reached. In clay products, the texture is habitually due to the gradual disappearance of the initial mineral grains into an indistinguishable matrix. Consequently, we get every variety of texture, as the initial grains vary from very coarse to impalpably fine, and as these grains are but little affected, or greatly affected, or wholly dissolved in the matrix. A third variation comes in from the fact that most silicate magmas tend to crystallize or become stony in cooling, and their crystallization may be of a coarse type visible to the eye, or so fine as to require the microscope to detect it.

Many people are misled by the fact that a fairly well vitrified clay, if cooled very suddenly, by withdrawal from the hot kiln, shows a glassy or smooth fracture on breaking. This is because it did not have the usual time to crystallize. In thin sections, under the microscope, every burnt clay shows abundant glassy material surrounding the residual undissolved grains. Sometimes it will show crystals, which have separated out of the fused matter in cooling. But to the eye, normally cooled clays very rarely look glassy, even when very far along in the process of vitrification. The normal microscopic texture of a well-vitrified clay shows abundant pieces of the initial mineral grains remaining, but firmly bound and cemented into a strong body by a matrix usually glassy in character, but sometimes crystallized quite thoroughly. The type of the claybody, whether natural or artificial, naturally imposes wide differences in this respect. One would not expect a piece of fine porcelain and a paving brick to show an equally fine texture, though each may be almost equally well along towards viscosity. It is therefore not an easy task to so accurately define the texture of vitrification, as to

make it possible to use it quantitatively in the decision of cases. It offers useful assistance to any single observer dealing with one product, but like the color test it cannot be applied by different observers to different products in any but the most general way.

Hardness.—The exact measurement of the quality known as hardness is another matter requiring the best endeavors of the well-trained and well-equipped physicist, and is wholly beyond the reach of the people who have to render practical judgments on vitrification. Simple little make-shift methods of determination, which can be applied by untrained workers are highly inexact. The arbitrary scale of mineral hardness, adopted by Moh, is of use to clayworkers only between points 6 and 7, namely, the hardness of feldspar and the hardness of quartz, because in that range practically all glasses and fused artificial silicates fall. The hardness of tempered steel, which of itself varies considerably, is in the same general position in the hardness scale as the glasses. We may therefore classify the hardness of burnt clays as being above steel, or equal to steel, or below it, etc., but here again the standard is very crude and inexact, and the test depends upon the steel used and the force used to apply it, as well as the thing tested.

There is another reason why the hardness of clayware is difficult to measure, namely, the fact that it is in almost every case a heterogeneous body. As stated elsewhere, there are usually grains of the initial minerals, in every stage of solution into an incorporating glass-like or slag-like matrix, and so the steel knife may cut one mineral grain, slip over the next, etc., leaving the operator unable to decide whether he has cut the test piece or not. In fact, cutting a clay product means usually dislodging or breaking pieces loose from their cementing matrix. When the vitrification is just begun, the grains break loose easily, that is, the clay cuts. But as the proportion of silicate mixture grows, and the grains which remain are themselves hardened more and more by heat, the knife leaves less and less impression, until it slides harmlessly over the smooth surface of a perfect glass, or is worn off in a lead-like streak of metal on the vitrified surface.

Still further, vitrified clays are invariably bubbly, or full of gas cells, or blebs, for reasons to be discussed later. These

gas cells may be microscopic, but if they become large enough to be visible, the thin partitions between adjoining cells easily break down under the pressure of a vigorously applied steel blade. A clay body which is spongy from over-fire will often cut, while a dense body containing usually less glassy matrix and not so many gas cells, will resist the blade. Some clays never become dense enough to resist the knife well at any commercial range of temperature, owing to lack of easy fusing constituents to form the cementing material. Nevertheless, such clays may become spongy through over-fire, whenever the heat becomes excessive for such fused materials as have formed.

The hardness test is therefore always bound to be a very crude indeterminate method of judging vitrification, and very difficult of standardization. It is, however, so simple and so easy to get roughly comparative data by the use of a knife, that it will always be used by individuals for their own convenience. It does not offer any hope of giving a standard method of measuring vitrification.

Strength.—The strength of clays shows the same progressive change as the other properties do, but the strength maximum is by no means always coincident with the most perfect vitrification. By strength, we mean the ability of the clay to resist disruption under stress, or friction, or both. The resisting power of a clay is by no means equally good under all the different kinds of stress which may be applied. Some resist crushing well; others show a high modulus under the cross-breaking or beam test; others resist impact well; and others again will resist rubbing and friction. One single clay is not apt to give a high rating in all these tests. It is common, therefore, to test a product by that form of strength test which lies closest to its intended use. The factors, elasticity, hardness, homogeneity, freedom from internal stresses, freedom from structural flaws, as well as mere cohesion, all go to make up the strength of the clay, and every change in burning temperature affects all of these factors, some for better, some for worse. The strength is therefore the resultant of these factors. Better vitrification improves one factor at the expense of another. In general, the best physical strength, at least by some methods of testing, is found before the best vitrification of a clay is reached.

The strength test in any form therefore cannot be recommended as a universal or general mode of testing vitrification.

Specific Gravity.—Density, in the sense of the true specific gravity, is a function of the minerals of which a clay is composed. If heavy minerals like iron oxide, calcite, hornblende, etc., are abundant, at the expense of lighter minerals like kaolinite and quartz, the specific gravity will naturally be high, and vice versa. The true specific gravity of a raw and burnt clay varies only by small amounts. It is true that some of the component minerals are subject to small changes in specific gravity under the influence of heat; quartz, for instance, may be converted into chrystoballite, a lighter mineral, etc., but these changes are usually negligible. The true specific gravity is wholly useless as a mode of measuring vitrification, and the apparent specific gravity, while useful to the chemist or clayworker as a mode of following the structure changes of a given clay at relative stages of vitrification, is of no use for establishing an absolute standard.

Compactness.—The degree of solidity or compactness of the mass as a whole, or the ratio between the solid matter and the voids, furnishes the best basis for estimating the progress of vitrification in a clay, or the degree of vitrification attained in any individual piece of clayware. The causes of the changes in compactness in a clay under vitrification are several. They must be dealt with under the following heads:

1. Obliteration of the communicating void system, existing between the initial grains.

2. Obliteration of the laminations and structure marks left in the clay by the process of molding or forming.

3. Formation of new cavities by the evolution, imprisonment and expansion of the gases in the mass.

From what has been already set forth, the gradual condensation of the clay mass as the burning progresses would be expected. The solution of the grains in each other enables closer contact than is possible between independent solid masses. In the case of those grains not dissolved, the matrix encompasses it more closely than other solid grains could do.

In the dried clay, before burning, no matter whether the clay be molded by dry pressing, or stiff mud, or soft mud, or fluid casting, the mineral grains are not in perfect contact. The

voids in dry clays usually are less than in dry sand, because the small grains to some extent fill the space between the larger particles, but still measure close to 30 per cent.

The amount of the water used in forming, influences the voids or communicating pore system also. In general, the voids are supposed to represent the space between the grains when in the closest possible contact, but in some processes of manufacture, the excess water unquestionably takes up room, which is not recovered by the clay particles as they pull together in drying, so that the clay is made more porous thereby. The pores, or communicating voids of a dry clay make the specific gravity of the mass as a whole much lower than the mean true specific gravity of its mineral components.

Now, as the heat advances in burning, this pore system is obliterated. The mass shrinks, pulls together, and the air in the voids tends to escape. This fire shrinkage is a notable amount, often 25 or 30 per cent of the total volume. The clay must therefore gain in apparent specific gravity, as the same amount of mineral matter is crowded into less and less space.

The obliteration of structure marks caused by the flow of the clay in forming is usually very much less complete or satisfactory than the closing up of the voids. The laminations and cavities are much larger and their walls are too far apart to feel the pull of molecular attraction, and the surface tension of the minute quantities of fused silicate material is too high to enable them to bridge wide cracks. For this reason, large laminations are seldom obliterated, though many minor ones doubtless are. Also, the larger cavities usually form receptacles for the accumulation of gases which are being expelled from the neighboring material, and this gas tends to hold the cavity open or to enlarge it.

Mechanical pressure, caused by the weight of superimposed material, is probably the chief factor in closing up laminations in most claywares. When the mass is just beginning to show viscosity, pressure would undoubtedly tend to compress the walls of cavities which lie at right angles to the line of pressure. But, also, in cavities lying parallel to the line of pressure, it may open them out equally well. Bricks compressed to less than normal thickness, but with correspondingly increased cross-section, are not uncommon.

In opposition to these forces, which tend to solidify and increase the compactness of the clayware, is the action of the evolved, contained and imprisoned gases. It matters little whether the gas is imprisoned air which is left in the void spaces and sealed in by advancing vitrification, or whether it is carbonic acid, sulfuric acid, or oxgyen, set free by the breakdown of minerals or their interaction. The latter sources, when they are active, usually create much larger volumes of gases than the sealed pores contain, and hence lead to worse distortion of the body, but the physics of the process is the same, no matter what the source of the gas. As the vitrification advances, the mass more and more takes on the properties of a liquid, and the gas exerts more and more pressure as the temperature rises. Each little gas cavity tends to become a spherical bubble, no matter what its initial shape might have been. A microscopic cross-section of any clay which has begun to vitrify shows millions of these little spherical gas-blebs. The further along in the process, the more numerous and larger the bubbles become. Naturally, these gas-blebs tend to swell the mass. It is exactly the same action as yeast in dough.

We now see that in a vitrifying clay two forces are at work on its volume, one tending to cause shrinkage and greater compactness, the other tending to cause bloating and a frothy structure. In the early stages of vitrification, the shrinkage force exceeds in vigor the bloating force, and the clay grows denser. After a longer or shorter period of heat treatment, the two forces balance each other for a time, during which the volume remains constant. This period of constant volume may cover a very short temperature range, or quite a long one. Clays which have a long period of substantially constant volume in burning are said to have a wide vitrification range, and are in great demand.

In some clays, the ratios of whose mineral mixture are favorable to practically complete fusion, the swelling goes on extraordinarily fast, when once a certain point of liquidity is reached, and the clay may become a sponge having several times its initial volume. In others, where the amount of mineral matter tending to form the silicate matrix of the clay is small, and the amount of coarse material for this matrix to dissolve is large, the bloating effect progresses as gently and slowly as the

shrinkage did earlier in the heat treatment. But whenever and however it occurs, bloating always indicates over-fire and incipient fusion, and marks the passage of the best stage of vitrification. Some clays are still commercially salable for quite a time after the point of minimum volume and maximum compactness have been passed. But in general, they fall in strength very rapidly and in other desirable properties also.

Between these two types of voids, those left between the original grains, and those caused by the formation of sealed gas cavities, there is one great difference. The former creates a freely communicating capillary system, readily permeable to water, while the latter is non-communicating, and not permeable at all except as the walls of the gas cells may be ruptured in expansion or contraction.

The measurement of the extent to which the communicating capillary system still exists affords the best indication of the degree to which vitrification has progressed. This is tested by measuring the absorption, or amount of water absorbed by this communicating capillary system. Nearly all clay products absorb 20 per cent or more of their weight of water when burnt only to the early stages of vitrification. As the vitrification proceeds, the compactness increases, and the percentage of absorption decreases. The absorption does not usually come to a complete stop at any stage of the heat treatment, but reaches a minimum and then slowly increases again, due to the frothy or bloating condition of over-fired clay, into which some water always does penetrate. In very fine clay bodies, the perfection of the vitrification may be so great at the best stage that the absorption is virtually *nil*, and recourse is had to the use of ink or colored fluids to see if any signs of absorption at all can be noted. Allowance must be made in such cases for surface adhesion of the coloring liquid.

Other Properties.—The dielectric resistance of burnt clay is another mode of studying vitrification. It depends upon the same conditions as the absorption—in large part at least—and requires much more expensive methods of testing. Similarly, the translucence, or transmission of light through the body is to some extent an index of vitrification. The heat conductivity also improves with the compactness and decreases with the

spongy structure. Frost resistance is also a function of the dense structure. None of these, or other properties of the burnt clay, have any greater value in setting a standard of vitrification than the absorption test.

Reviewing the ground for a moment, we see that color, texture, hardness, strength and specific gravity are all affected in the heat treatment of clay, and that all may to some extent act as rough gages or modes of measuring the progress of vitrification. We see that color goes through a progressive series of changes, that the texture becomes finer and closer, that the hardness, the strength and the specific gravity all increase, but that exact measures of color, texture, hardness, strength or specific gravity are all impossible or costly, and cannot be correlated exactly with the progress of vitrification. On the other hand, we find that compactness is the one property which most clearly typifies the vitrification process, and that the absorption test makes an excellent and easily applied method of determining it. The absolute degree of compactness of a product is indicated by a single test. By a series of determinations made upon test pieces drawn at different temperatures, the progress of the vitrification process in a burn can be studied equally well by this simple method.

THE PERMISSIBLE ABSORPTION LIMITS.

Speaking of vitrification in its physical sense, and accepting the statement that the absorption test is the best measurable index for it, what limits should be set? Obviously, the answer to this question depends chiefly upon what one expects to do with the clay product in question. Vitrification is not an absolute term, and the whole matter comes down to a question of what degree of vitrification is needed.

There are three principal purposes or objects whose attainment is sought in making a vitrification requirement in a clayware. These are (1) frost resistance, or durability to weather; (2) impermeability to prevent absorption of colorants or dirt; and (3) dielectric strength, or insulating value. It is also required sometimes as a guarantee of strength and hardness, but, as already pointed out, it is not an accurate index of these properties, and if they are the properties sought, it is better to

specify directly in terms of those properties. Of course, vitrified wares are presumptively both hard and strong wares, and in a non-critical sense, the vitrification requirement may be considered as covering those properties.

The resistance of different types of claywares to frost and weather varies quite a good deal, owing to the influence of the process of manufacture. But in general, danger from frost may be said to be past when the absorption falls as low as five per cent. Instances of wares whose absorption has been lower than five per cent clearly failing to withstand weather have occurred very seldom in the observation of the writer, and in his opinion are rare. When found, the explanation should be sought in unusual structural conditions such as severe laminations, large flaws close to the surface, cracks permitting influx of considerable water at one place, or similar defects.

Engineers and others in writing specifications to safeguard clayware from weather failure have usually been much too severe in the limits assigned. Limits of one to two per cent are usual, and the limits of above three per cent are rare. Yet experience and tests have shown that the point of failure lies much higher and is usually above six per cent for stiff-mud wares and twelve per cent for soft-mud wares. The argument for a factor of safety which engineers customarily employ in their work applies here to a limited extent only, for in confining wares to a one-per-cent or two-per-cent limit to secure frost resistance, the factor of strength and resistance to wear has often been sacrificed, or heavily invaded, and in dealing with some clays at least, a certain loss of quality in the latter respect ought not be risked for the very doubtful gain in the former.

In cases where impermeability is the quality sought, the absorption must be very severely restricted or reduced to zero. For table wares, the power to absorb dirty or greasy water, either through cracks in the glaze or from areas of the body exposed by chipping, means stains which are ineradicable. Even slight absorption will soon leave its mark in wares which are exposed to washing several times a day, or several times per meal as in hotels. The same arguments patently apply to sanitary and toilet fixtures for interior use.

In attempting to safeguard wares for such uses, the specifica-

tions have usually insisted upon no change in weight after prolonged soaking in water. If this requirement is administered with discretion, and due consideration is given to getting the exterior of the test specimen wholly free from adhering surface water before taking the weight after immersion, this requirement has not proven difficult to meet. But, in order to make it still more searching it has frequently been specified that ink, or a colored solution, must not leave a stain when applied to the fractured surface of the ware. This makes a condition virtually impossible of fulfilment, for the adhesion of some coloring matter on the surface will always take place, so that a colored spot persists after repeated washing in water. The case of an appropriate solvent, such as oxalic acid, or a stronger one, will usually take away this adhering material promptly if it is on the surface, but much more slowly if the stain has really penetrated the pore system of the body. Hence, where stains applied on the raw fracture are made a part of the absorption test, the use of superficial solvents should also be made a part of the proof of actual penetration, otherwise the test is prohibitive and good wares will be rejectable at the caprice of the inspecting officer. For safeguarding the insulating quality of porcelains for high-tension service, the requirement must be extraordinarily rigid. No precaution which will accomplish the end sought can properly be omitted, and so far as the writer is informed, complaints on unreasonable severity of absorption tests do not come from the makers of this class of ware.

For low-tension wares for common interior wiring for 110 or 220-volt service, the application of the rigid requirements necessary in high-tension work seems unreasonable and unduly restrictive. Any porcelain maker *can* make absolutely non-absorptive ware. But he probably cannot make all of his ware absolutely non-absorptive, especially to the ink test, without measures which must raise the price of his product very materially. The question is one that cuts the public more than the porcelain maker. Should prices be held far higher in order to make ware stand a strict non-absorption test, when the conditions of use make the factor of safety enormously high without it?

The same logic also applies to the specifications for conduits

for street cable work, where a good strong tile with smooth skin is the needed qualification, and the lead-covered cable is itself the best safeguard against dampness.

In the three types of uses just discussed, commercial practice has already to some extent undertaken the problem of framing specifications. In the case of paving bricks, to some small extent in building bricks, in floor tile, roofing tile, sewer pipe, conduits, chemical stoneware, pottery of various grades, specifications founded on more or less knowledge of the product have become so common as to meet little opposition. Further, studies have been made and are being made constantly, developing our knowledge of the exact requirements upon which it is necessary to insist to safeguard the ultimate consumer and work no injustice upon the producer. It is the function of this Society, and particularly of its Committees C-3, C-4, C-6, C-8 and C-10, to continue and extend this useful service.

But in the fields already indicated and in many allied branches of clay manufacture, large quantities of material are still sold under specifications which are not very intelligent upon the vitrification requirement. It is very common indeed to find in specifications, a clause stating that the ware must be "vitrified," or "well vitrified" or "thoroughly vitrified," with no mention of any measurable property by which the vitrification can be proved or disproved. It is to reach cases of this class that this paper has been written.

The following case came up several years ago in the practice of the writer:

A company manufacturing large drain tile, chiefly for county ditch work, and which had behind it a number of years of successful operation, and whose product, laid in many miles of ditches, had given excellent service for years under all sorts of conditions, suddenly found itself dispossessed of contracts in the home county, where the natural market almost wholly lay, by the vitrification requirement. The specifications said the ware must be "vitrified." Virtually their entire product had been sold on this same specification for years past. No engineer, county commissioner, or rival bidder had ever raised the issue as to whether their ware was vitrified.

A new bidder in the field, coming in with a shale pipe of

unusually dense and smooth fracture, raised the point. The engineer in charge, a new man in the position, examined by mere visual inspection the competing tiles. The home material was made from a glacial clay, which contained a good deal of rather coarse gritty mineral matter which could not be absorbed by the silicate matrix in any reasonable burning treatment, and still showed as spots in the fracture. The fracture was stony and somewhat granular. The absorption was from two to four per cent, the majority under three per cent. The strength was good and the quality of its performance under conditions of use was known to be excellent, after years of trial. And yet, on the ground that the home product was not vitrified, he ruled it out and bought a tile whose fracture was more nearly glassy, but whose weather resistance was no better and whose strength was less, especially in regard to brittleness and inability to endure shocks and strains.

The above case is typical of many, and shows the need of an interpretation of the word "vitrified" when used with no explanatory phrases or criteria of measurement. Undoubtedly every kind of clay product, wherever its vitrification is of importance, should be carefully safeguarded by standard specifications. This will be attained in time, and these specifications will fluctuate from relatively high absorption in some types to none at all, not even ink stains in others.

But until the true conception of vitrification becomes practically universal, and the present looseness in the term can be eradicated, a definition of vitrification, or the adjective "vitrified," to apply in all specifications where the term is not otherwise defined, should be adopted by this Society, for the guidance of all contestants, in court or out. For such a definition the writer proposes the following:

PROPOSED DEFINITION OF THE WORD "VITRIFIED".

Where the word "vitrified" is used in a contract or agreement in describing the properties of a clay product, and is not further defined in the contract, it shall mean that the product shall have attained in the firing process such a degree of compactness that its mean absorption does not exceed three per cent of its own initial weight of pure water, when subjected to

complete immersion in cold water for forty-eight hours, or boiling water for three hours. Not less than three tests shall be made in determining the mean absorption, and no single test may exceed the mean by more than 0.5 per cent. Neither the possession of the visual properties or appearance of a glass, nor any definite degree of physical strength, hardness, color, or specific gravity, shall be implied as an attribute of vitrification.

[EDITOR'S NOTE.—Following the presentation of this paper at the annual meeting, the definition of the word "Vitrified" was, on motion, referred to Committee C-3 on Standard Specifications for Brick, with instructions to confer with the other technical committees dealing with clay products.]

DISCUSSION.

Mr. Schuyler.

Mr. Mont Schuyler.—I believe that particular emphasis should be placed on the fact that this definition is intended to hold only when there is no other definition included in a specification for vitrified material. It is well known that many paving blocks absorb as high as 5 per cent of moisture, although they may be most excellent material and may have proved satisfactory in every way in service.

A Member.

A Member.—I should like to ask if it is the intention that the amount of absorption as proposed here is to be determined on a finished piece, covered, as I suppose it may be, with a partial layer of glaze, or if it is the intention that broken pieces of the article shall be examined, which may, perhaps, absorb more?

Mr. Orton.

Mr. Edward Orton, Jr.—Replying to that question, I would say that it is undeniably preferable that the measurement should be made upon the whole piece, but that in many instances, to exact this requirement, would involve a good deal of difficulty and perhaps some hardship in the transportation or immersion and weighing of large pieces of ware. I cannot too strongly emphasize the fact that this definition is only intended to cover cases which no one else has taken the trouble to cover. I think sewer-pipe men, brick men, porcelain men, in fact everybody that has any occasion to use clay products accurately, should define what absorption they consider to represent the proper compactness for that particular ware. But, where use of the word "vitrified" is made in specifications, without taking the trouble to explain what it means, I think that the making of an absorption limit, arbitrarily, as proposed in my paper, is necessary. The test should be made with whole ware, if possible, and broken ware, if necessary. I think that the use of such a limit as 3 per cent will be found to fairly meet most cases.

Mr. Camp. Mr. A. D. Camp.—It seems to me that in order to avoid misunderstandings the procedure governing this absorption test should be more definitely described. After the immersion in water, is the water clinging to the surface of the sample removed before weighing, and if so by what means? A variation in this procedure would have a very material effect upon the magnitude of the absorption determination.

A FUNGUS BED TEST OF WOOD PRESERVATIVES.

By Cloyd M. Chapman.

SUMMARY.

A general description is given of a method of test which was prepared in the laboratory of Westinghouse Church Kerr and Co. for the purpose of gathering data on the relative values of wood-preservative materials when applied by several methods to different kinds of wood. The specimens are exposed in a fungus bed out of doors and taken up and examined periodically.

The materials used include foreign and domestic coal-tar creosotes, water-gas creosotes, wood-tar creosotes, mineral oil, vegetable and animal oils and inorganic salts in solution.

The methods of application are as follows: cold dip; hot dip; and continued boiling, followed by cooling in the preservative.

The specimens, 12 in. in length, are placed upright in a fungus bed with 3 in. above ground and 9 in. below ground. The fungus bed is prepared by mixing a quantity of pulverized sheep manure with screened loam.

A FUNGUS BED TEST OF WOOD PRESERVATIVES.

By Cloyd M. Chafman.

Introduction.

The test described in this paper was drawn up in the testing department of Westinghouse Church Kerr and Co. for the purpose of conducting a series of tests to gather data as to (1) the class or kind of materials commonly used as wood preservatives which gives the best protection to wood, partly embedded in damp soil and partly exposed to the air, (2) the method of applying the preservative which will give satisfactory results, and (3) the kinds of wood which will take the different methods of treatment most effectively.

The test described was chosen after considering the methods in use in other laboratories, and after consulting with many persons connected with the wood-preserving industry. The details of the test are therefore simply a collection of the suggestions made by many people and no claim whatever is made for novelty or unusual merit. It is simple and easy to carry out and if experience should show that the results obtained agree with those obtained in actual service, under analogous conditions, then the test will be of value to those interested in accelerated tests.

The nature of the exposure is similar to that of fence posts, telegraph and telephone poles, wooden sleepers or sills laid in contact with damp ground, and similar exposures. It is hardly to be expected that the value of the preservatives when used under other and different classes of exposure can be safely judged by the results of this test.

Treatment of Specimens.

Apparatus Used.—For the purpose of treating the wood specimens quickly and conveniently a series of four small cylindrical metal containers are arranged as shown in Fig. 1. These containers are of galvanized iron and just large enough to

easily take in four specimens at a time. The containers are arranged on a common support with a gas burner under each and a thermometer hook over each. They are placed at a small angle with the vertical to facilitate a more uniform distribution of heat to the contents by means of convection currents. A rack is arranged conveniently near on which the specimens after treatment are hung up to drain and dry.

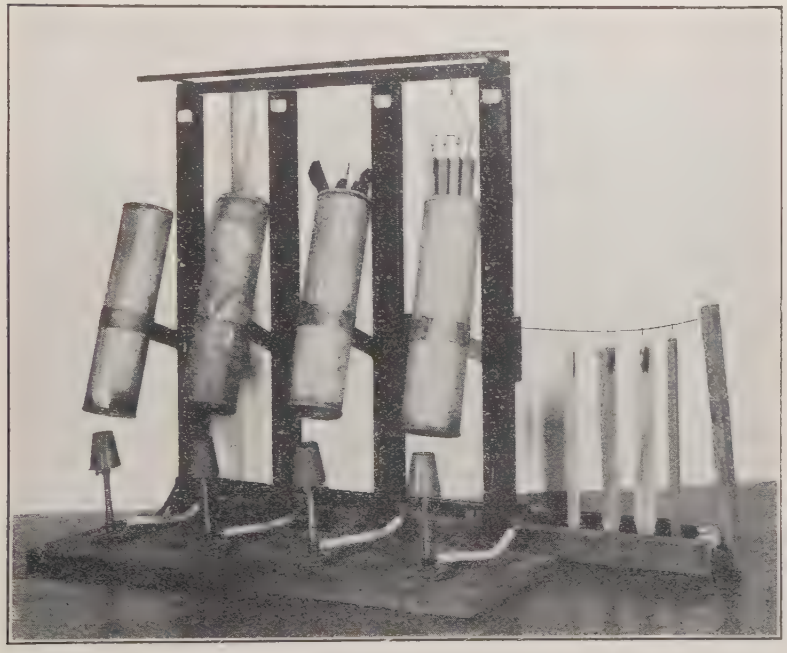


FIG. 1.—Apparatus Used for Treating Specimens.

A small wire nail is inserted in the end of each piece of wood and bent over to form a hook. A wire lifter is provided of such shape that four wood specimens may be hung on it by means of the hooks just mentioned and the specimens will then hang about $\frac{1}{4}$ in. apart. By this means the specimens may be dipped in the preservative simultaneously and without touching one another. The treatment is therefore equal on all four sides of each specimen. As the wood specimens would float in the preservatives,

a weight is provided on the top of the specimens sufficient to submerge them.

Methods of Treatment.—Three methods of treatment are used, and are chosen to represent three methods commonly used in commercial work, namely, cold dip, hot dip, and saturation. The cold-dip treatment is considered about equivalent to a brush coat with cold materials. The specimens are immersed for 15 seconds in the cold liquid and then hung up to allow the surplus to drain off. The temperature of the preservative for this treatment is about 21° C.

The hot-dip treatment is deemed equivalent to the hot open-tank treatment frequently used for the treatment of wood in the field. The specimens are immersed for one minute in the hot liquid and then hung up to drain. The temperature of the preservative for this treatment is about 100° C., except in those cases in which the preservative boils or gives off vapors at this temperature. In such cases the temperature is reduced only enough to prevent boiling. These are the solutions of inorganic compounds and one or two low-boiling distillates.

The saturation treatment is intended to produce about the same degrees of saturation in these small specimens that the closed-tank pressure treatment would produce in full-size timbers. The specimens are kept immersed in the hot liquid for one hour, allowed to cool in the liquid and then hung up to drain. The temperature of the preservative for this treatment is maintained at 105° C., except when the preventative boils at a lower temperature, in which cases the temperature is kept just below the boiling point of the liquid. Because of the comparatively small size and thickness of the specimen it is probable that something approaching the full-cell treatment results from this method.

Woods Treated.—Several varieties of wood should be chosen for the test, and they should be representative of the woods commonly used in the section of the country in which the preservatives would be used. Specimens of each of the woods are sawed, but not planed, approximately to the dimensions $\frac{1}{2}$ in. thick, $1\frac{1}{2}$ in. wide and $\frac{1}{2}$ in. long. One end of each specimen is tapered to a point. The sawed specimens are thoroughly air-dried before treatment.

Preservatives Used.—A wide variety of materials may be used as preservatives in the treatment of the specimens, such as the products of coal tar, water-gas tar, and wood tar; also animal or vegetable oils, solutions of inorganic compounds, etc. These materials are obtained from the manufacturers or dealers, or made up in the laboratory.

The samples submitted by manufacturers should be from the regular market grade of each of the products.

Records Kept.—The data recorded in the laboratory are as follows:

1. A serial number for each preservative, for convenience only.

2. The trade name of each preservative or a descriptive name.

3. The name of the manufacturer or dealer from whom the sample was obtained.

4. The chemical and physical characteristics of the preservative in sufficient detail to identify it, including specific gravity, distillation test of creosotes, etc.

5. The weight of each specimen of wood shortly before treatment.

6. The weight of each specimen after treatment and after the surplus preservative has been allowed to drain off the surface. From these weights taken before and after treatment, the quantities of preservative used per square foot and per cubic foot of specimen are computed.

DESCRIPTION OF TEST.

Exposure.—The treated specimens are exposed in a fungus bed out of doors. This bed is prepared by digging out a pit of suitable area about 18 in. deep, screening the dirt through a $\frac{1}{4}$-in. screen to remove the roots, stones, etc., thoroughly mixing with the screened dirt a quantity of dried and pulverized sheep manure, then replacing the mixture in the pit.

This method of accelerating the action of the fungi by the use of sheep manure was decided upon because of the very rapid rotting of wood that had been noted in soil where sheep manure had been used as a fertilizer. In fact, by the use of this material it seems to be possible to accelerate the decay of wood to almost

any degree desired. In the present method it is not deemed advisable to accelerate the action to any very great extent and for that reason only a small amount of the sheep manure was added to the soil. A small quantity of decayed wood was also added to the soil of the fungus bed.

The specimens are inserted for a depth of about 9 in. in this bed, leaving about 3 in. projecting above the surface of the soil. The distance between specimens is about 3 in.

Fig. 2 shows the specimens in the fungus bed.

Examination and Rating.—Every six months after exposure the specimens are all removed from the bed, brushed off with a

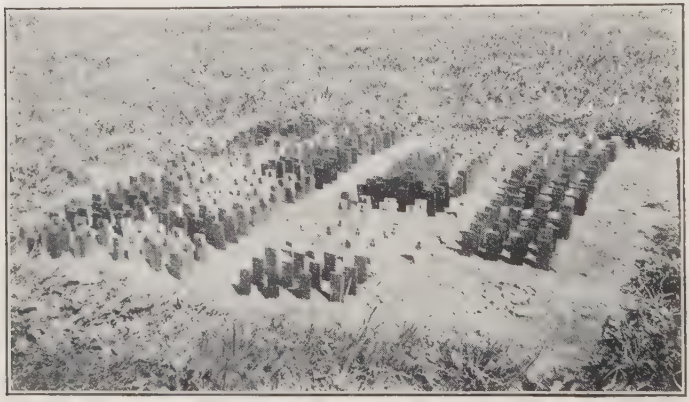


FIG. 2.—Specimens in Fungus Bed.

soft wire brush and examined for decay. Softening of the surface is taken as the guide to decay and the finger nail serves as the means of testing.

The scale of ratings used is entirely arbitrary and comparative. A rating of 100 indicates that no sign of decay is apparent. Ninety-five indicates a slight softening of the surface or edges, and so on down the scale until zero indicates a complete rotting through, so that the wood may be easily crushed or broken with the fingers. Record is also made of any checking or cracking of any specimen. Photographic records are kept of the condition of typical specimens.

The fungus bed is entirely dug out and rescreened and re-

mixed, and additional sheep manure added at the time of each examination. The specimens are then returned to the bed and left undisturbed until time for the next examination.

CONCLUSION.

In conclusion, it may be well to emphasize the following points in connection with this method of test:

1. It is an accelerated test. The degree of acceleration is dependent on the quantity of stimulant mixed with the soil in the bed.

2. It gives information as to the behavior of the preservatives in only one class of exposure; namely, the class in which the wood comes into contact with damp ground.

3. The results obtained are purely comparative, as no attempt is made to control conditions of exposure such as humidity, temperature, etc.

4. The details of the test are simple and easy to carry out, and the small amount of apparatus required is very inexpensive so that almost anyone interested may carry out the tests.

SOME EXPERIMENTS ON TECHNICAL BITUMENS.

By S. R. Church and John Morris Weiss.

SUMMARY.

The object of the investigations here described was to afford a comparison, by means of laboratory analyses, of eighteen varieties of asphalts and tar pitches; and to subject test specimens of the same materials to exposure tests. These were as follows:

1. Effect of overheating;
2. Effect of gas drip;
3. Effect of illuminating gas;
4. Effect of one year's exposure to sunlight and air.

The procedure and results of these exposure tests are fully described.

Additional exposure tests, including effect of oxygen and nitrogen gas at 212° F., and effect of water, dilute acid and dilute alkali solutions, are described, but the results are not given because of incompleteness. Further investigation along these lines is contemplated.

It is shown:

1. That a moderate degree of overheating does not seriously affect the bitumens tested;

2. That gas drip attacks all the bitumens tested, but that some resist its action for a longer time than others;

3. That in an atmosphere of illuminating gas, at 60° F. for six days, some of the bitumens are measurably softened;

4. That after exposure to sunlight and air for one year, while the exposed surface of the bitumens tested has manifestly undergone marked change as evidenced by photographs of these surfaces, analyses of the test specimens show only slight physical or chemical changes.

SOME EXPERIMENTS ON TECHNICAL BITUMENS.

By S. R. Church and John Morris Weiss.

About two years ago we obtained authentic samples of typical bitumens, and undertook a series of laboratory tests on these materials. Some of these tests are completed, others are still in progress. It is the purpose of this paper to describe the materials as received; their preparation or modification for testing; the tests so far completed, and a brief reference to others now in progress, or contemplated. It is hoped that the paper may be useful in affording a comparison of a rather wide range of tar pitches and asphalts, by the usual methods of proximate analysis, supplemented by a number of tests designed to determine the effect on these bitumens of exposure to various conditions.

The Materials Tested.

1. *Origin.*—The bitumens tested were as follows:

1. Pitch from gas-house tar (horizontal retorts);
2. Pitch from gas-house tar (inclined retorts);
3. Pitch from gas-house tar (vertical retorts);
4. Pitch from coke-oven tar (Otto-Hoffman type);
5. Pitch from coke-oven tar (Semet-Solvay type);
6. Pitch from water-gas tar (Eastern);
7. Pitch from water-gas tar (Central);
8. Pitch from blast-furnace tar (Scotland);
9. Pitch from pine-tar (Florida);
10. Stearine pitch (tallow pitch);
11. Asphalt, refined Bermudez, fluxed;
12. Asphalt, refined Trinidad, fluxed;
13. Grahamite (Oklahoma), fluxed;
14. Gilsonite (Colorado), fluxed;
15. Asphalt (California), straight-run;
16. Asphalt (Mexican), straight-run;
17. Asphalt (Texas), blown, fluxed;
18. Asphalt (Texas), straight-run, fluxed.

For the purpose of these experiments it was decided to bring all the test specimens to a uniform consistency of 40-deg. (4 mm.) penetration. To this end the tar pitches were prepared in the laboratory by distilling crude tar in a copper still of the usual type, until the residue had the desired consistency. The native asphalts and two residual asphalts were fluxed, as hereafter described.

Table I gives an index to the character of the crude tars used. Table II gives proximate analyses, as received, of the asphalts which required softening, and also the proximate analysis of the Mexican flux oil used in reducing asphalts

TABLE I.—TESTS OF CRUDE TARS.

Tar No.	Kind of Tar.	Specific Gravity at 60° F.	Free Carbon, per cent.
1	Gas-house (horizontal retorts)	1.27	28.9
2	Gas-house (inclined retorts)	1.24	24.3
3	Gas-house (vertical retorts)	1.15	4.0
4	Coke-oven (Otto Hoffman)	1.18	9.8
5	Coke-oven (Semet-Solvay)	1.19	10.8
6	Water-gas (Eastern)	1.08	0.1
7	Water-gas (Central)	1.11	0.4
8	Blast-furnace (Scotland)	1.15	15.9
9	Pine (Florida)	1.06	0.03

Nos. 11, 12, 13, 14, 17 and 18 to the desired consistency. In the preparation of these fluxed asphalts, due precautions were taken against overheating. The finished materials were passed while hot through a 10-mesh sieve, and preserved in tightly closed, friction-top cans.

2. *Proximate Analysis.*—The methods followed in comparing the crude tars, the asphalts as received, the flux oil, and in the final comparison of test specimens, were as follows:

(*a*) Solubility in carbon bisulfide by the method of the Office of Public Roads;[1]

[1] Hubbard and Reeve, "Methods for the Examination of Bituminous Road Materials," *Bulletin No. 38*, Office of Public Roads, pp. 27–29.

(*b*) Solubility in benzol and toluol, by the method described by S. R. Church;[1]

(*c*) Solubility in carbon tetrachloride, by the method of the Office of Public Roads;[2]

(*d*) Fixed carbon and ash, by a modification of the method of the Joint Committee on Coal Analysis of the American Society

TABLE II.—TESTS OF ASPHALTS AS RECEIVED.

Asphalt No.	Kind of Asphalt.	Insoluble in Carbon Bisulfide, per cent.	Fixed Carbon, per cent.	Ash, per cent.	Melting Point, deg. Fahr.	Penetration at 77° F. with 100 g. for 5 seconds, deg.	Specific Gravity at 60° F.
11	Bermudez, refined...	7.8	14.7	3.3	144	19	1.08
12	Trinidad, refined....	44.0	10.8	39.0	...	..	1.31
13	Grahamite.....	2.9	50.3	2.3	...	..	1.11
14	Gilsonite.....	0.2	13.8	0.3	...	..	1.04
17	Texas blown.....	0.9	17.1	0.3	170	10	1.06
18	Texas straight run...	1.1	18.1	0.6	170	17	1.11

NOTE.—The proximate analysis of the Mexican flux oil is as follows:

TEST OF FLUX OIL.

	PER CENT.
Specific gravity at 60° F	1.01
Loss by evaporation 7 hrs. at 400° F., per cent	7.7
Flash point, deg. Fahr	464
Fire point, deg. Fahr	495
Paraffin scale, per cent	2.5
Insoluble in 88° Baumé naphtha, per cent	20.5
Soluble in carbon bisulfide, per cent	99.9

for Testing Materials and the American Chemical Society, as described by S. R. Church;[3]

(*e*) Specific gravity, by the "platinum-pan" method, as described by J. M. Weiss;[4]

[1] "Methods for Testing Coal Tar, etc.," *Journal of Industrial and Engineering Chemistry*, Vol. 5, No. 3, p. 195 (1913).

[2] *Ibid.*, p. 31.

[3] "Methods for Testing Coal Tar, etc.," *Journal of Industrial and Engineering Chemistry*, Vol. 3, No. 4, p. 227 (1911).

[4] "Specific Gravity—Its Determination for Tars, Oils and Pitches," *Journal of Industrial and Engineering Chemistry*, Vol. 7, No. 1, p. 21 (1915).

(*f*) Melting point in air, by the cube method, as described by S. R. Church;[1]

(*g*) Penetration by the standard Dow machine.

TABLE III.—PROXIMATE ANALYSES OF BITUMENS PREPARED FOR EXPOSURE TESTS.

Bitumen No.	Kind of Bitumen.	Insoluble in Benzol and Toluol, per cent.	Insoluble in Cold Carbon Bisulfide, per cent.	Insoluble in Carbon Tetrachloride, per cent.	Melting Point (air), deg. Fahr.	Specific Gravity at 60° F.	Fixed Carbon, per cent.	Ash, per cent.	Penetration at 32° F. with 200 g. for 1 min., deg.	Penetration at 77° F. with 100 g. for 5 seconds, deg.	Penetration at 115° F. with 50 g. for 5 seconds, deg.
1	Pitch from gas-house tar (horizontal retorts)	34.9	34.9	38.4	125	1.30	41.5	0.2	2	39	Too soft
2	Pitch from gas-house tar (inclined retorts)	30.8	32.0	34.8	123	1.28	37.0	0.2	2	40	"
3	Pitch from gas-house tar (vertical retorts)	8.0	10.3	15.6	125	1.19	16.3	0.2	2	44	"
4	Pitch from coke-oven tar (Otto Hoffman type)	21.1	19.7	27.1	126	1.25	28.5	0.4	3	41	"
5	Pitch from coke-oven tar (Semet-Solvay type)	18.3	17.4	23.3	126	1.25	28.2	0.1	2	39	"
6	Pitch from water-gas tar (Eastern)	5.0	2.0	9.1	125	1.18	24.8	0.2	3	39	"
7	Pitch from water-gas tar (Central)	7.8	1.5	11.9	124	1.19	25.3	0.0	3	41	253
8	Pitch from blast-furnace tar (Scotland)	28.4	30.0	32.6	135	1.23	14.4	11.8	8	41	324
9	Pitch from pine tar (Florida)	7.8	4.6	11.2	127	1.13	19.9	0.1	3	41	Too soft
10	Stearine pitch (tallow pitch)	....	....	....	209	1.02	8.8	1.9	21	42	81
11	Asphalt, refined Bermudez, fluxed (16.3 % oil)	5.6	5.5	5.7	145	1.07	11.6	3.5	8	41	235
12	Asphalt, refined Trinidad, fluxed (46.5 % oil)	22.4	24.4	21.8	149	1.21	9.3	20.6	9	39	180
13	Grahamite (Oklahoma) fluxed (85.0 % oil)	0.0	0.12	0.3	191	1.04	16.8	0.0	13	41	70
14	Gilsonite (Colorado), fluxed (76.0 % oil)	0.06	0.25	0.3	153	1.02	11.5	0.0	10	41	176
15	Asphalt (California), straight-run	0.3	0.5	0.4	153	1.03	13.0	0.2	12	40	149
16	Asphalt (Mexican), straight-run	0.5	0.3	0.4	150	1.04	15.8	0.0	10	41	136
17	Asphalt (Texas), blown, fluxed (27.7 % oil)	1.3	1.2	1.0	150	1.04	13.5	0.4	10	41	130
18	Asphalt (Texas), straight-run, fluxed (12.7 % oil)	1.8	1.3	1.7	137	1.04	13.3	0.4	7	41	261

All results represent averages of at least two determinations, and the penetration tests represent averages of six determinations.

[1] "Methods for Testing Coal Tar, etc.," *Journal of Industrial and Engineering Chemistry*, Vol. 5, No. 3, p. 195 (1913).

Table III[1] gives a comparison of the bitumens as prepared for these experiments.

EXPERIMENTS TO COMPARE THE EFFECT OF EXPOSURES.

EFFECT OF OVERHEATING.

It was decided to subject the bitumens for two hours in an open vessel to a temperature at least 50° F. in excess of the maximum temperature usually permitted when instructions are given regarding the melting of pitch or asphalt for paving or waterproofing purposes.

(*a*) *Description of Test.*—An open iron crucible (Eimer and Amend catalogue No. 2879) of approximately 60-cc. capacity, was used. Fifty grams of pitch or 45 g. of asphalt were taken in each case, assuming that these quantities represented approximately equal volumes. The pitches were heated for two hours at 400° F. and the asphalts for two hours at 450° F.

(*b*) *Result of Tests.*—In Table IV is given the loss in weight after cooling, and the hardening of the bitumens, as expressed by penetration at 77° F., compared with original penetration, and by increase in melting point.

It may be noted that the pitches, excluding the blast-furnace-tar pitch and pine-tar pitch, show an average loss of 6.2 per cent compared with an average loss of 3.8 per cent for the asphalts, and that the pitches average 24° F. increase in melting point compared with an increase for the asphalt of 20° F. It may also be noted that the pitch of highest free-carbon content had a much lower loss and showed less hardening, than the average pitches. Of the asphalts, Bermudez, Trinidad and California were the most susceptible, and Mexican and Texas

[1] Since the preparation of this paper, the authors' attention has been called to the fact that the characteristics of the straight-run California asphalt used in these tests, as set forth in Table III, are not exactly in accordance with the usual characteristics of that product, particularly with reference to its susceptibility. We are informed on the highest authority that straight-run California asphalt having a penetration of 40 at 77° F. should show a penetration of more than 200 at 115° F. Our attention has also been directed to the fact that Texas residual products, both blown and straight-run, can be had in any desired consistency, and that it might have been better had we obtained these products at a consistency of 40-deg. penetration, instead of fluxing harder products. It can only be said that in obtaining the asphalts, we ordered commercial products through recognized manufacturing or distributing agencies and in the case of the residual materials we stated that we desired products of 40-deg. penetration.

products least so. The results indicate that moderate overheating has no serious effect on any of the bitumens tested.

EFFECT OF EXPOSURE TO GAS DRIPS.

The material commonly known as gas drips is a condensate from illuminating gas, and is found to a greater or less degree

TABLE IV.—RESULTS OF OVERHEATING BITUMENS.

Bitumen No.	Kind of Bitumen.	Temperature of Heating for 2 Hours, deg. Fahr.	Net Weight, g.	Loss in Weight, per cent.	Melting Point in Air. deg. Fahr.		Penetration at 77° F. with 100 g. for 5 seconds, deg.	
					Before.	After.	Before.	After.
1	Pitch from gas-house tar (horizontal retorts).	400	50	2.2	125	136	39	17
2	Pitch from gas-house tar (inclined retorts)...	400	50	4.8	123	151	40	6
3	Pitch from gas-house tar (vertical retorts)...	400	50	9.6	125	159	44	2
4	Pitch from coke-oven tar (Otto Hoffman type)	400	50	6.8	126	144	41	3
5	Pitch from coke-oven tar (Semet-Solvay type)	400	50	5.8	126	143	39	3
6	Pitch from water-gas tar (Eastern).....	400	50	6.8	125	161	39	5
7	Pitch from water-gas tar (Central).....	400	50	7.6	124	151	41	3
8	Pitch from blast-furnace tar (Scotland).....	400	50	6.4	135	171	41	6
9	Pitch from pine tar (Florida).....	400	50	4.0	127	133	41	14
10	Stearine pitch (tallow pitch).....	450	45	1.3	209	207	42	30
11	Asphalt, refined Bermudez, fluxed.....	450	45	8.0	145	163	41	8
12	Asphalt, refined Trinidad, fluxed.....	450	45	5.1	149	181	39	11
13	Grahamite (Oklahoma), fluxed.....	450	45	3.3	191	217	41	20
14	Gilsonite (Colorado), fluxed.....	450	45	3.8	153	172	41	20
15	Asphalt (California), straight-run.....	450	45	6.2	152	175	40	13
16	Asphalt (Mexican), straight-run.....	450	45	0.7	150	169	41	20
17	Asphalt (Texas), blown, fluxed.....	450	45	1.1	149	143	41	26
18	Asphalt (Texas), straight-run, fluxed.....	450	45	2.2	136	164	41	21

in all gas mains. From leaky mains this liquid, as well as the gas itself, seeps through the earth, and sometimes forms a source of destruction to bituminous materials used in waterproofing foundations, subways, etc. Gas drip has proved to be an effective solvent for most bitumens, although it does not, as has

been erroneously stated, consist largely of benzol, but contains higher boiling hydrocarbons.

(*a*) *Description of Test.*—For this experiment we adapted glass tubes specially constructed for another purpose. A piece of paper perforated with pin holes was sealed over the flanged opening of the tube and a film of bitumen was folded over the flange and sealed with melted pitch. The film of bitumen backed by perforated paper covered the flanged end of the tube. This film was prepared as follows:

Heavy drawing paper was coated with a sugar-dextrin solution, dried, and folded box-shape with the coated side out. This box was dipped into the melted bitumen, drained for a few seconds, and opened out. Ordinary manila paper, with a similar dried sugar-dextrin coating, was placed with the coated side against the bitumen and the film between the two papers rolled while still slightly warm, with a piece of $\frac{3}{4}$-in. glass tubing. The edges were trimmed off and the film between the papers soaked in cold water until the sugar-dextrin had dissolved, leaving the bitumen film completely detached from the papers. The separated film was then trimmed to the proper size. The films were 0.012 to 0.019 in. thick. Only those that were free from visible imperfections were tested.

A sample of the gas drips used gave results as follows:

TEMPERATURE, DEG. CENT.	BULB DISTILLATION, PER CENT.
100	1
120	11
140	35
160	58
180	77
200	87
220	92
Specific gravity at 60° F	0.897

The tubes were immersed in a beaker of gas drips, to give a pressure of 1 in. of liquid on the bitumen film. The time in minutes for penetration of the film was noted.

(*b*) *Result of Tests.*—Table V gives the time in minutes for the failure of the bitumen films. All the films were pervious, but the time of failure varied widely. The asphalts and the

water-gas-tar pitches were more rapidly attacked than the coal-tar pitches. Stearine pitch showed rather surprising resistance.

EFFECT OF EXPOSURE TO ILLUMINATING GAS.

Attention has recently been drawn to this subject by a paper dealing with "The Effect of Leaking Illuminating Gas on

TABLE V.—PERMEABILITY OF BITUMEN FILMS TO GAS DRIPS.

Bitumen No.	Kind of Bitumen.	Time to Fail, minutes.
1	Pitch from gas-house tar (horizontal retorts)	135
2	Pitch from gas-house tar (inclined retorts)	120
3	Pitch from gas-house tar (vertical retorts)	34
4	Pitch from coke-oven tar (Otto Hoffman type)	95
5	Pitch from coke-oven tar (Semet-Solvay type)	99
6	Pitch from water-gas tar (Eastern)	3
7	Pitch from water-gas tar (Central)	5
8	Pitch from blast-furnace tar (Scotland)	8
9	Pitch from pine tar (Florida)	7
10	Stearine pitch (tallow pitch)	105
11	Asphalt, refined Bermudez, fluxed	4
12	Asphalt, refined Trinidad, fluxed	3
13	Grahamite (Oklahoma), fluxed	14
14	Gilsonite (Colorado), fluxed	4
15	Asphalt (California), straight-run	2
16	Asphalt (Mexican), straight-run	14
17	Asphalt (Texas), blown, fluxed	2
18	Asphalt (Texas), straight-run, fluxed	1

Bituminous Pavements."[1] This paper has been criticized by the President of the American Gas Institute;[2] nevertheless, illuminating gas can and does sometimes exert a destructive action on bitumens used in the construction of city pavements, and also on bituminous water-proofing materials.

(*a*) *Description of Test.*—A special apparatus was designed, as shown in Figs. 1, 2 and 3. The bitumens were weighed into

[1] G. C. Warren, *Proceedings*, Am. Soc. Munic. Imp., Boston, 1914.
[2] E. C. Jones, *Gas Institute News*, February, 1915.

porcelain combustion boats, and placed in the iron tubes. The temperature was maintained within one degree of 60° F., by passing cold water continuously through the coil, and at the same time having a burner under the apparatus, controlled by a Roux bi-metallic regulator. Gas was passed through the tubes at the rate of about 3.5 cu. ft. per hour, the rate of flow controlled by

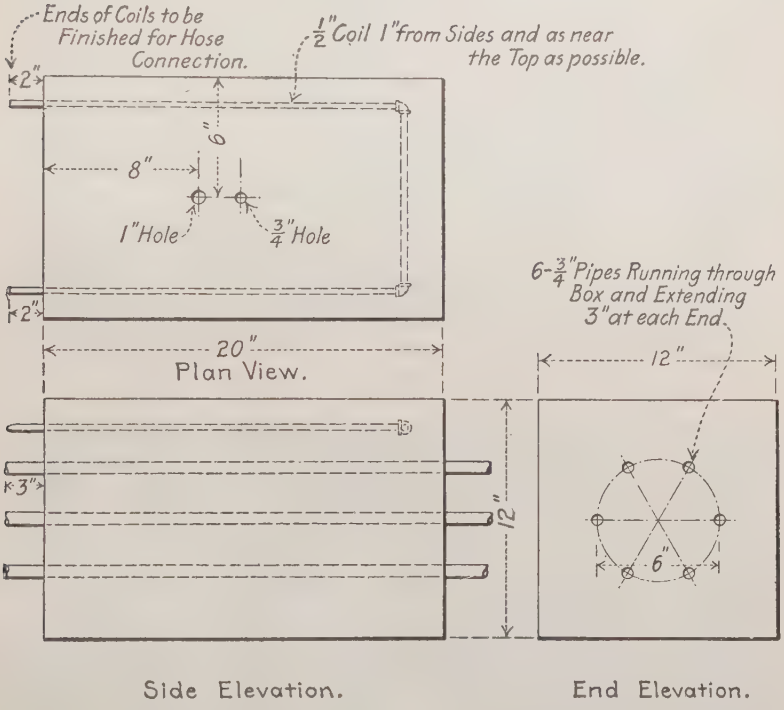


FIG. 1.—Galvanized-Iron Box Used for Illuminating-Gas Exposure Tests on Bitumens.

$\frac{1}{64}$-in. openings in plugs on the exit ends of the tubes. The bitumens remained in the gas for six days, and then were removed and weighed. Each bitumen was then poured into a small container, and its penetration at 77° F. determined.

(*b*) *Result of Tests.*—Table VI shows the effect on the bitumens of this exposure, as expressed by gain in weight and

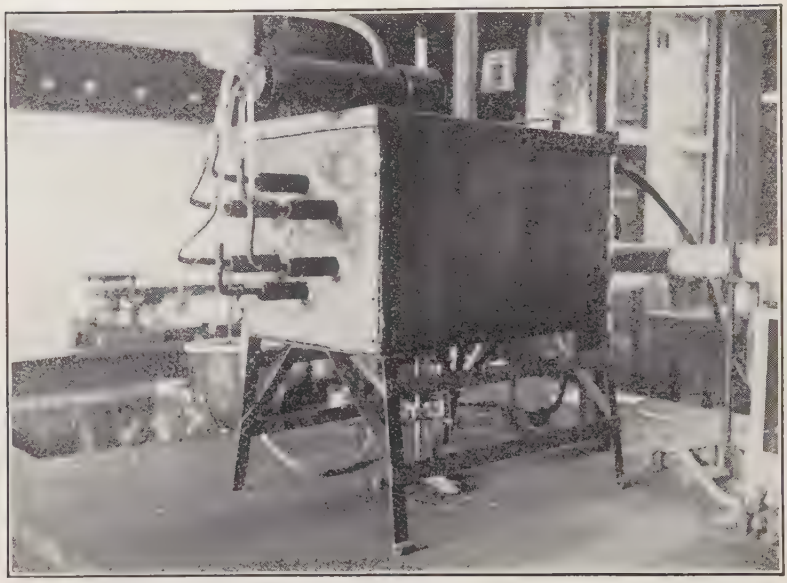


FIG. 2.—Illuminating-Gas Test Box.

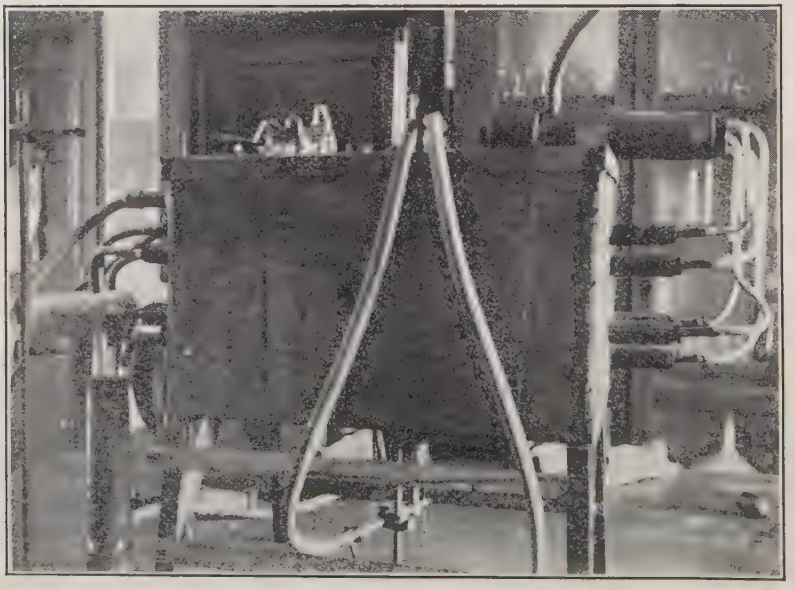


FIG. 3.—Illuminating-Gas Test Box.

by softening. It may be noted that the effect on the bitumens varied considerably. An appreciable softening is exhibited by some of the asphalts, and the water-gas-tar pitches.

EFFECT OF EXPOSURE TO SUNLIGHT AND AIR.

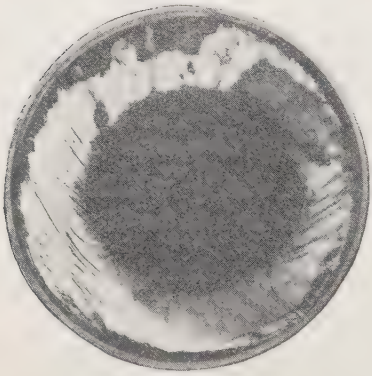
This experiment followed somewhat closely the lines of Hubbard and Reeves' work.[1] The significance to be attached

TABLE VI.—EFFECT OF EXPOSURE TO ILLUMINATING GAS.

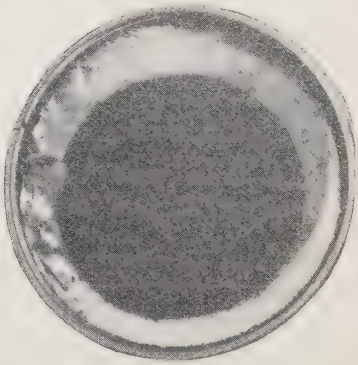
Bitumen No.	Kind of Bitumen.	Gain in Weight, per cent.	Penetration at 77° F. with 100 g. for 5 seconds, deg.	
			Before.	After.
1	Pitch from gas-house tar (horizontal retorts)	0.2	34	38
2	Pitch from gas-house tar (inclined retorts)	0.2	37	41
3	Pitch from gas-house tar (vertical retorts)	0.0	38	37
4	Pitch from coke-oven tar (Otto-Hoffman type)	0.2	41	42
5	Pitch from coke-oven tar (Semet-Solvay type)	0.0	31	28
6	Pitch from water-gas tar (Eastern)	1.9	30	71
7	Pitch from water-gas tar (Central)	1.3	38	76
8	Pitch from blast-furnace tar (Scotland)	0.1	36	36
9	Pitch from pine tar (Florida)	1.2	45	74
10	Stearine pitch (tallow pitch)	2.1	38	50
11	Asphalt, refined Bermudez, fluxed	2.2	41	85
12	Asphalt, refined Trinidad, fluxed	1.5	36	56
13	Grahamite (Oklahoma), fluxed	2.5	42	63
14	Gilsonite (Colorado), fluxed	1.8	43	66
15	Asphalt (California), straight-run	3.8	39	96
16	Asphalt (Mexican), straight-run	2.4	31	56
17	Asphalt (Texas), blown, fluxed	2.1	42	74
18	Asphalt (Texas), straight-run, fluxed	2.4	42	78

to results of such exposure tests is open to discussion. If exposed to direct action of rain and wind, the results might be quite different. Manifestly, however, no comparison of weight, consistency, insoluble matter, etc., between different test specimens, would be possible under the latter conditions of exposure.

[1] Hubbard and Reeve, "The Effect of Exposure on Bitumens," *Journal of Industrial and Engineering Chemistry*, Vol. 5, No. 1, p. 15 (1913).

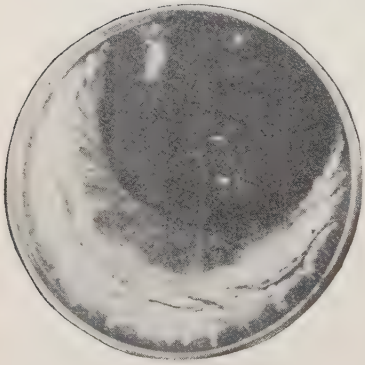


Pitch No. 1.



Pitch No. 2.

FIG. 4.



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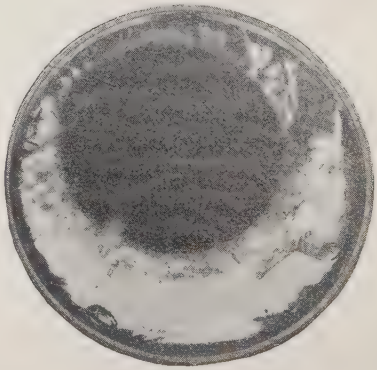
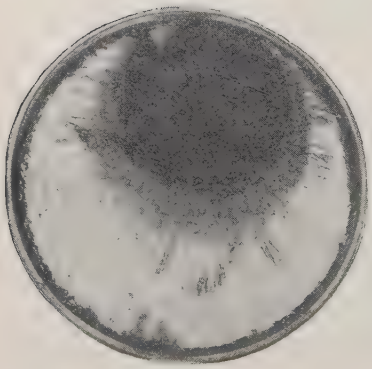


Pitch No. 4.

FIG. 5.



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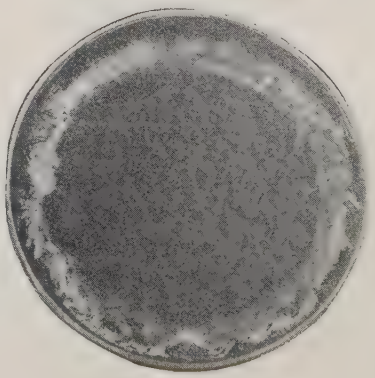
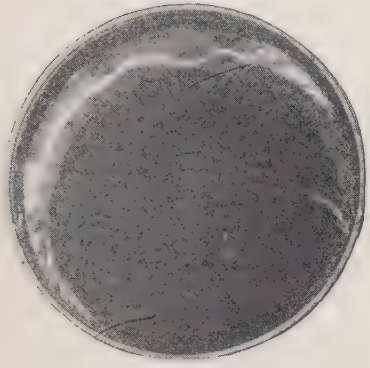


Pitch No. 6.

FIG. 6.



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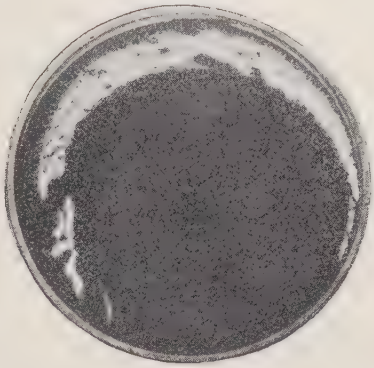
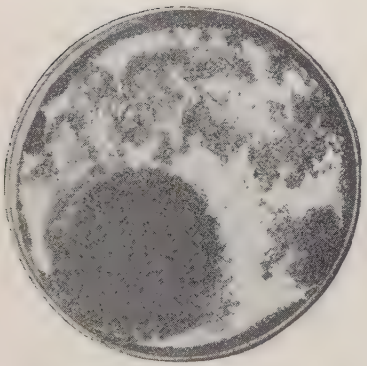
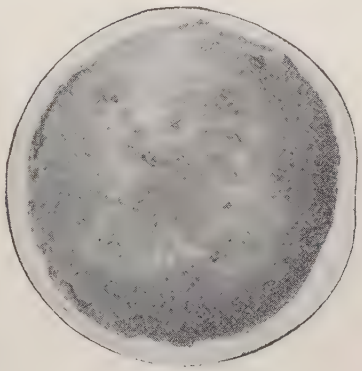


Pitch No. 8.

FIG. 7.

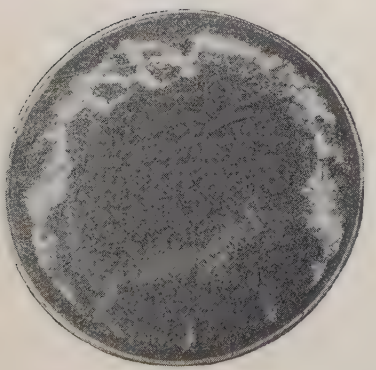


Pitch No. 9.

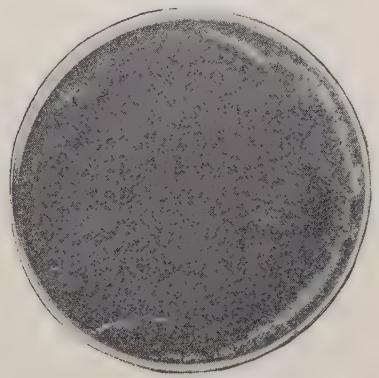


Pitch No. 10.

FIG. 8.

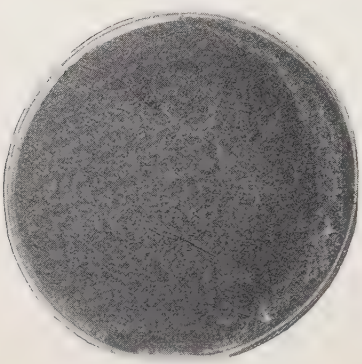


Asphalt No. 11.



Asphalt No. 12.

FIG. 9.



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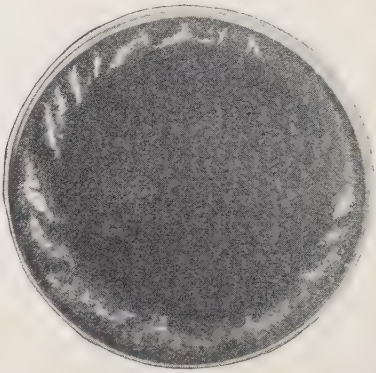
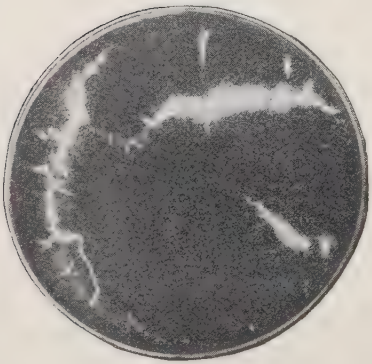


Asphalt No. 14.

FIG. 10.



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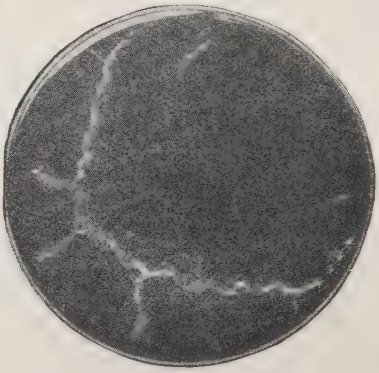
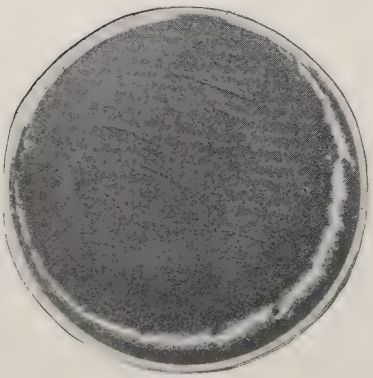


Asphalt No. 16.

FIG. 11.



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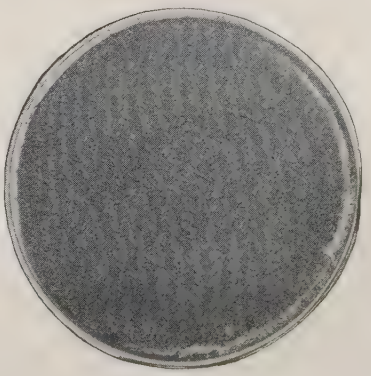


Asphalt No. 18.

FIG. 12.

(*a*) *Description of Test.*—The bitumens were exposed in petri dishes, each holding a layer approximately 5 mm. in depth. The dishes were placed in a dustproof box with glass top. Air circulation was provided by means of side vents filled with cotton and protected from rain by a dashboard. The test specimens remained in this box on the laboratory roof for a period of one year; they were then removed and tested.

TABLE VII.—APPEARANCE OF BITUMENS AFTER EXPOSURE OF ONE YEAR TO SUNLIGHT AND AIR.

Bitumen No.	Kind of Bitumen.	Color of Streak.	Nature of Surface.
1	Pitch from gas-house tar (horizontal retorts)	Black	Hard
2	Pitch from gas-house tar (inclined retorts)	Black	Hard
3	Pitch from gas-house tar (vertical retorts)	Dark brown	Hard
4	Pitch from coke-oven tar (Otto-Hoffman type)	Black	Hard
5	Pitch from coke-oven tar (Semet-Solvay type)	Black	Hard
6	Pitch from water-gas tar (Eastern)	Resinous brown	Soft
7	Pitch from water-gas tar (Central)	Dark rusty brown	Soft
8	Pitch from blast-furnace tar (Scotland)	Brown	Hard
9	Pitch from pine tar (Florida)	Dark brown	Hard
10	Stearine pitch (tallow pitch)	Brown	Rubbery
11	Asphalt, refined Bermudez, fluxed	Brown	Leathery
12	Asphalt, refined Trinidad, fluxed	Brown	Medium
13	Grahamite (Oklahoma), fluxed	Ochre	Leathery
14	Gilsonite (Colorado), fluxed	Ochre	Leathery
15	Asphalt (California), straight-run	Yellow	Medium
16	Asphalt (Mexican), straight-run	Ochre	Medium
17	Asphalt (Texas), blown, fluxed	Brown	Medium
18	Asphalt (Texas), straight-run, fluxed	Brown	Medium

(*b*) *Result of Tests.*—The appearance of the bitumens after one year's exposure is shown in Figs. 4 to 12, inclusive. The color of the streak of the central oxidized portion, and nature of the surface when scratched with a knife, are noted in Table VII.

Table VIII gives the comparative proximate analyses of the bitumens before and after one year's exposure. It may be noted that the changes in the bitumens are not very marked, either in chemical or physical properties. In consistency, the seven tar pitches show an average increase in melting point of

TABLE VIII.—COMPARATIVE PROXIMATE ANALYSES OF BITUMENS BEFORE AND AFTER EXPOSURE OF ONE YEAR TO SUNLIGHT AND AIR.

Bitumen No.	Kind of Bitumen.	Thickness, mm.	Total Insoluble in Benzol, per cent.		Total Insoluble in Carbon Tetrachloride, per cent.		Fixed Carbon, per cent.		Ash, per cent.		Specific Gravity at 60° F.		Melting Point in Air, deg. Fahr.		Penetration at 77° F. with 100 g. for 5 seconds, deg.		Penetration at 32° F. with 200 g. for 1 minute, deg.	
			Before.	After.	Before.	After.	Before.	After.	Before.	After.	Before.	After.	Before.	After.	Before.	After.	Before.	After.
1	Pitch from gas-house tar (horizontal retorts)	5.0	34.9	35.4	38.4	44.1	41.5	43.6	0.2	0.3	1.30	1.30	125	126	39	23	2	2
2	Pitch from gas-house tar (inclined retorts)	4.8	30.8	31.1	34.8	39.6	37.0	39.7	0.2	0.2	1.28	1.29	123	128	40	19	3	2
3	Pitch from gas-house tar (vertical retorts)	4.7	8.0	8.9	15.6	16.2	16.3	19.3	0.2	0.2	1.19	1.22	125	132	44	11	3	2
4	Pitch from coke-oven tar (Otto-Hoffman type)	4.6	21.1	21.1	27.1	29.3	28.5	30.1	0.4	0.4	1.25	1.25	126	127	41	21	3	2
5	Pitch from coke-oven tar (Semet-Solvay type)	5.3	18.3	20.4	23.3	23.8	28.2	29.6	0.1	0.1	1.25	1.27	127	126	39	16	2	3
6	Pitch from water-gas tar (Eastern)	5.0	5.0	5.1	9.1	11.0	24.8	26.9	0.2	0.2	1.18	1.19	126	127	39	25	3	2
7	Pitch from water-gas tar (Central)	5.1	7.8	7.4	11.9	11.0	25.3	27.0	0.0	0.2	1.19	1.21	124	128	41	19	4	1
8	Pitch from blast-furnace tar (Scotland)	5.1	28.4	29.0	32.6	32.4	14.4	17.7	11.8	11.9	1.23	1.23	136	142	41	19	8	4
9	Pitch from pine tar (Florida)	4.9	7.8	8.9	11.2	10.6	19.9	22.5	0.1	0.1	1.13	1.15	127	132	41	22	4	3
10	Stearine pitch (tallow pitch)	4.3	....	....	....	....	8.8	12.5	1.9	2.1	1.02	1.03	209	282	42	14	21	5
11	Asphalt, refined Bermudez, fluxed	5.0	5.6	5.8	5.7	4.9	11.6	13.8	3.5	3.7	1.07	1.07	145	147	41	26	8	4
12	Asphalt, refined Trinidad, fluxed	4.9	22.4	22.0	21.8	24.2	9.2	10.5	20.6	20.4	1.21	1.22	149	156	39	17	10	8
13	Grahamite (Oklahoma), fluxed	5.0	0.0	0.2	0.3	0.8	16.8	18.8	0.0	0.1	1.04	1.04	191	223	41	12	13	4
14	Gilsonite (Colorado), fluxed	5.0	0.1	0.4	0.3	0.5	11.5	13.5	0.0	0.1	1.02	1.02	153	170	41	19	10	7
15	Asphalt (California), straight-run	4.3	0.3	1.9	0.4	0.8	13.0	15.1	0.2	0.2	1.03	1.04	153	166	40	12	12	5
16	Asphalt (Mexican) straight-run	5.2	0.5	1.0	0.4	0.4	15.8	17.4	0.0	0.1	1.04	1.04	150	172	41	14	11	7
17	Asphalt (Texas), blown, fluxed	4.9	1.3	1.4	1.0	1.3	13.5	16.7	0.4	0.5	1.04	1.04	150	164	41	15	11	6
18	Asphalt (Texas), straight-run, fluxed	5.1	1.8	2.0	1.7	1.5	13.3	16.1	0.4	0.4	1.04	1.05	137	153	41	19	7	6

3° F., and the eight asphalts an increase of 16° F., while the pitches show a decrease in penetration at normal temperature of 21 deg., and the asphalts a decrease of 23 deg. None of the bitumens shows any measurable increase in matter insoluble in benzol, but some of the pitches and one asphalt have increased 2 to 6 per cent in matter insoluble in carbon tetrachloride.

EXPERIMENTS IN PROGRESS AND CONTEMPLATED.

EFFECT OF EXPOSURE TO ATMOSPHERES OF OXYGEN AND NITROGEN FOR 144 HOURS AT 212° F.

It was suggested by Mr. Herbert Abraham and others that the apparent volatile matter given off on heating bitumens at elevated temperatures in an air oven, was undoubtedly not a true measure of volatility, because bitumens under such conditions may at the same time give off volatile oils and take up oxygen from the air. Mr. Abraham suggested that the true measure of the volatility of bitumens might be learned by determining the difference between loss in an atmosphere of illuminating gas at a given temperature, and loss or gain in an atmosphere of oxygen at the same temperature. We are conducting tests along these lines, using nitrogen as an inert gas in preference to illuminating gas.

(*a*) *Description of Test.*—Gas from cylinders is passed through a gas meter, dried and purified by passing through a tower containing solid caustic potash, a wash bottle containing a solution of 20-per-cent caustic potash, a wash bottle containing concentrated sulfuric acid, and a tower containing anhydrous calcium chloride. The gas then enters a glass tube in which the test specimens are placed. The tube is surrounded by a steam jacket, by means of which the interior temperature is maintained at 212° F. with a variation not exceeding 0°.5 F. Duplicate test specimens consisting of 8 g. each, in weighed porcelain boats, are placed in the tube and remain for six days in a current of gas flowing about $\frac{1}{3}$ cu. ft. per hour. On removal and after cooling, loss or gain in weight, and penetration at 77° F. are determined.

(*b*) *Result of Tests.*—All the bitumens lose weight after exposure for six days at 212° F. in an atmosphere of nitrogen, the losses varying from 0.1 to 9 per cent. All the bitumens show

less loss after similar exposure in oxygen, in some cases showing a slight actual gain in weight. The difference between loss in nitrogen and loss in oxygen may be taken to represent gain from oxidation. This varies from less than 0.1 to 4.5 per cent. We have not presented in detail the results obtained from this test, because to us the results indicate the necessity of conducting additional experiments before drawing conclusions.

EXPOSURE OF THIN BITUMEN FILMS TO DILUTE ACID, DILUTE ALKALI AND WATER.

Believing that it might be possible, by exposing thin films of bitumen to the effect of water, acid and alkaline solutions, to determine in a reasonable time of exposure what would be the probable result of continued exposure to less severe conditions, the following method was devised:

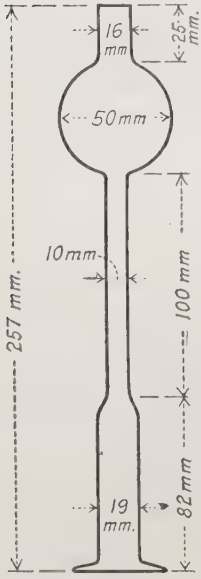


FIG. 13.—Tube for Bitumen Film Tests.

(*a*) *Description of Test.*—Special glass tubes (Fig. 13) were designed for this test. The films were prepared as described under the heading "Effect of Exposure to Gas Drips," and they were approximately 0.015 in. thick over the opening of the tube. Freshly dehydrated copper sulfate was placed in each tube to a depth of about 1 in. on the surface of the perforated paper. The bulb of the tube was filled with anhydrous calcium chloride, the tube plugged with cotton, and a capillary tube inserted. The flanged ends of the tubes were immersed in water, 2.5-per-cent sulfuric-acid solution, and 2.5-per-cent sodium-hydroxide solution, to a depth of 1 in. Failure of a film is at once indicated by a blue coloration of the copper sulfate.

(*b*) *Result of Tests.*—The result of this test to date is not sufficiently concordant to justify recording, but the test is described in the hope that suggestions may be received, leading to its modification and improvement. The acid and alkaline solutions have been increased in strength to 20 per

cent, and 10 per cent, respectively. Dilute alkaline solutions seem to be more destructive to bitumens than dilute acid solutions.

TESTS CONTEMPLATED.

The tests contemplated are as follows:

(*a*) Exposure to light and air on a larger scale and for a longer period than before, having enough test specimens of each bitumen so that samples can be removed for testing at intervals of one year, and so that more extended tests can be applied to the samples after long exposure.

(*b*) Exposure to oxygen and nitrogen under such conditions that larger test specimens can be used; and provision made for determination of water that may be formed during the oxygen exposure.

The authors do not desire to present any conclusions in connection with the experiments here recorded. It is hoped that the results in themselves may be sufficiently useful to those engaged in the study or use of the technical bitumens, to justify their presentation.

The authors wish to express their indebtedness to Messrs. Hubbard, Reeve, Abraham and Dow for suggestions that influenced the procedure. Much of the experimental work was conducted by Messrs. Downs and Dunning of our own laboratory, who also aided in designing special apparatus.

DISCUSSION.

Mr. Forrest. MR. C. N. FORREST (*presented in written form*).—It is not unusual in an attempt to assemble a collection of materials of a general class from several separate sources, for use in a series of experiments, to find that apologies or explanations are necessary as regards the representative character of some of them, before such experiments are completed. From the footnote on the sixth page of the paper, it is observed that the authors' experience has not been an exception in this respect.

Those who are engaged in the application of bituminous materials for paving and waterproofing purposes have probably observed that the escape of illuminating gas and gas drips from the service pipes in city streets has caused some local disturbance of pavements or waterproofing in a few instances, but the illuminating gas industry is not responsible for any great amount of damage on this account.

The authors have shown that all classes of bituminous materials experimented with are quickly dissolved or destroyed by illuminating gas and gas drips. In this respect there are scarcely more than microscopic differences between the various bituminous materials involved.

As representatives of the coal-tar industry the authors are doubtless well qualified to select representative types of pitch from the several producing sources of pitch. The types mentioned by them are perhaps recognized and sharply differentiated inside the coal-tar industry, but to the consuming public there are practically only coal-tar pitch and water-gas-tar pitch when pitch for any general purpose is purchased.

The results of the experiments do not, as just remarked, indicate much more than microscopical differences between the different pitches.

In regard to the asphaltic materials which have been included in the experiments, we find that apologies have already been made as to the representative character of some of them as technical bitumens.

Mr. Forrest.

Exception should also be taken to the type of flux oil used for softening the hard asphalts, etc. The oil used by the authors should only be regarded as a specific type of Mexican flux oil, and the bitumens which have been combined with it, only as specific types of fluxed bitumens. They are not technical bitumens of the type which their designation might indicate, and representative of what is commercially available under such designation.

A flux oil of greater fluidity than that employed by the authors, or one prepared from other crude petroleum than Mexican, is available, and, as a matter of fact, in much more general use in the asphalt industry than such heavy oil or flux as the latter.

The consuming public seldom discriminates as to the character of flux oil which the manufacturer of fluxed asphalt may employ, any more than it does as to whether the pitch desired shall have been produced from horizontal, vertical or inclined retort gas tar or one or more coke-oven tars.

Messrs. Church and Weiss.

MESSRS. S. R. CHURCH AND JOHN MORRIS WEISS (*Authors' closure by letter*).—In his discussion of our paper, Mr. Forrest seeks to minimize the importance and significance of the results set forth therein. The disintegrating effect of illuminating gas and gas drips upon bitumens used in the construction of pavements and sub-surface waterproofing is probably much more general and serious than is usually supposed. Since the paper was presented, our attention has been called to the instance of a sheet asphalt pavement in Washington, D. C., which has been re-laid at least three times within the past ten years, and whose failure in each case has been attributed to the effect of gas. One of the authors has recently examined numerous service cuts in sheet asphalt pavements, and in a surprisingly large number of cases the odor of gas was distinctly apparent in the cut pieces of the pavement.

In regard to the "microscopic" character of the differences between the various bituminous materials tested, we would call attention to Bermudez asphalt cement, which, after the exposure to illuminating gas, was softened from an original penetration of 41 to a penetration of 85, while Pitch No. 1 had an original penetration of 34 and a final penetration of 38. An engineer

Messrs. Church and Weiss.

ordering asphalt of 40 penetration, and receiving asphalt that tested 85, would hardly consider the difference "microscopic." Also, in the exposure to gas drip, a film of Trinidad asphalt cement, approximately $\frac{1}{66}$ in. thick, was permeated in three minutes, while a film of coal tar pitch of the same thickness required 100 minutes. Engineers interested in the waterproofing of tunnels under city streets may consider such a difference more than "microscopic."

Mr. Forrest says that the consuming public knows only coal-tar pitch and water-gas-tar pitch. He should, however, be aware of the fact that specifications for pitch for its various technical uses are quite as common as specifications for asphalt, and quite as restrictive.

With reference to the type of flux oil used, we do not presume to say which of the several types available are in most general use in the asphalt industry, but we know that large quantities of flux, such as that used in our experiments, are used in roofing manufacture, and we are informed that large quantities are also used in other branches of the asphalt industry. Opinions differ as to the desirability of using a more fluid flux oil, but it is unlikely that any one type of flux would meet with universal approval, or could be considered standard.

THE FUSIBILITY OF COAL ASH.[1]

By A. C. Fieldner, A. E. Hall and A. L. Feild.

SUMMARY.

The growing demand for the inclusion in coal specifications of the "fusing" or "softening" temperature of the ash as a safeguard against excessive clinker troubles, has led the Bureau of Mines to take up a study of laboratory methods of determining fusibility and the relation of these results to clinker formation under furnace conditions. A study was made of the principal causes for variations in the softening temperatures of coal ash as indicated by the deformation of Seger cones molded from the pulverized ash. Comparative tests were made on a series of 18 types of coal ash, in six different furnaces such as are in more or less common use for determining the degree of fusibility of silicate mixtures. It was found that the softening temperature of a given ash was influenced by the following factors:

1. Shape and size of cone;
2. Inclination of cone;
3. Fineness of ash;
4. Rate of heating;
5. Oxidizing or reducing nature of atmosphere in which the cone was heated.

The nature of the atmosphere had by far the largest influence on the softening temperature, causing differences as high as 400° C. in some cases. The highest results were obtained either in air with no reducing gases present, or in a strong reducing atmosphere whereby the iron oxides were reduced to metallic iron. The lowest results were obtained in such atmospheres which were able to reduce the iron oxide to the ferrous form but not to the metallic state.

[1] By permission of the Director, U. S. Bureau of Mines.

A new method for determining this lowest softening temperature was then developed, whereby the iron was caused to combine in the slag principally in the ferrous state, by heating the ash in an atmosphere of approximately equal parts by volume of hydrogen and water vapor. A comparison of analyses of actual clinker slags from boiler furnaces, and of the ash cones fused in the 50:50-per-cent hydrogen-water vapor mixture, showed the iron component in both cases to be principally in the ferrous state. Therefore the proposed fusibility test should have a closer relation to clinker formation than tests made in oxidizing atmospheres that produce slags in which the iron exists principally in the ferric state.

THE FUSIBILITY OF COAL ASH.

By A. C. Fieldner, A. E. Hall and A. L. Feild.

Introduction.

The growing demand for the inclusion in coal specifications of the "softening" or "fusing" temperature of the ash as a safeguard against excessive clinker troubles, has led the Bureau of Mines to take up a study of laboratory methods of determining fusibility and the relation of the results to clinker formation under practical furnace conditions.

The present paper gives a summary[1] of the results obtained in a study of the various factors which influence the softening temperature of ash when molded in the form of Seger cones; there is described also a new method of making the Seger-cone test, whereby the ash is caused to soften and form slags similar to those found in fuel-bed clinkers.

Necessity of Standardizing Fusion Tests.

Before any chemical or physical test can be prescribed for use in specifications, it must be so standardized that comparable results can be secured in different laboratories. Such standardization is a comparatively simple matter when the test involves the determination of fixed chemical or physical constants such as the percentage of sulfur in coal, or the melting point of pure copper. When, however, the test is not based on a fixed constant, all the known factors influencing the result must be studied and standardized, and even then the results may be subject to variations from unknown and uncontrollable factors. An example of an empirically standardized test, which can only be duplicated by a rigid adherence to a prescribed method of procedure, is the determination of the volatile matter in coal. The volatile matter is not in the coal as a definite compound, but

[1] The complete report of this investigation will be published in a Technical Paper of the Bureau of Mines.

is the result of decomposition by heat; the amount of volatile matter obtained varies with the temperature and the rate of heating.

NATURE OF SOFTENING-POINT TEST.

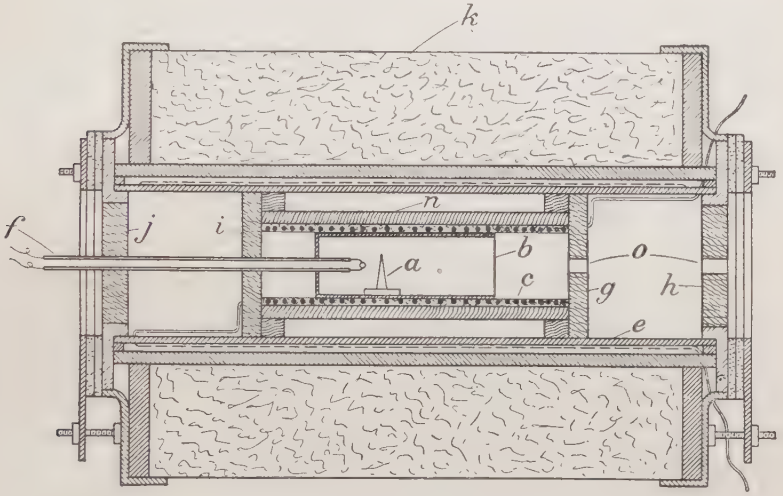
Determination of Softening Point.—The determination of the "fusing" or "softening" temperature of coal ash is in many respects analogous to the determination of volatile matter. Coal ash, being a complex mixture of various oxides and silicates, does not have a fixed, definite melting point where it changes from a solid to a liquid state. On slowly heating a sample of ash it successively shrinks, sinters and then gradually softens into a slag of diminishing viscosity. This range of visible softening may extend over 200° C., in the case of ashes which form rather viscous slags. Since there is no sharp change of state we can only choose empirically some point or temperature interval where the slag approximates a certain viscosity, and call this the softening temperature.[1] Usually this is done by molding the ash in the form of a small triangular pyramid similar to a Seger cone, and noting the temperature of deformation on heating the cone at a definite rate. The temperature at which the cone has bent over until the apex touches the base, or failing to bend has collapsed to a lump, is taken as the softening point.

Relation of Softening Point to Melting Point of Eutectic.—It is obvious then that the softening temperature of an ash cone does not represent a definite melting point, but rather the temperature at which some constituent or eutectic has melted in sufficient amount to allow the cone to collapse. As pointed out by Day and Shepard,[2] a cone made of silicate mixtures which are capable of forming eutectics, begins to weaken as soon as the eutectic begins to melt; its further progress is then governed entirely by the relative quantity of eutectic present and its viscosity after melting. If the constituents of the ash are such as

[1] The authors prefer the use of the term "softening temperature" rather than the more commonly used term "fusing temperature." Fusion point or fusion temperature implies that the substance melts at a definite temperature to the liquid state, which is far from the truth in the case of coal ash.

[2] "The Lime-Silica Series of Minerals," *American Journal of Science*, (4), Vol. 22, p. 267 (1906). Also, for a full discussion of the theory of Seger cones, see Sosman, "The Physical Chemistry of Seger Cones," *Transactions*, Am. Ceramic Soc., Vol. 15, pp. 482–498 (1913).

to form a relatively large proportion of thinly fluid eutectic, the softening point of the cone will lie close to the melting point of the eutectic; on the other hand, if there is a large excess of some refractory component as silica or aluminum silicate, or if the eutectic is viscous, the excess component will form a rigid skeleton which is not readily pulled down by the flowing eutectic. In the latter case the softening point will approach more nearly the melting point of the rigid component.



k, Heraeus tube furnace, type B; *e*, porcelain tube, $2\frac{5}{8}$ in. inside diameter, 12 in. long, wound with platinum-foil heating element; *n*, alundum tube, $1\frac{1}{2}$ in. inside diameter, 6 in. long, $\frac{3}{16}$-in. wall; *c*, 17 ft. of 0.03-in. diameter platinum wire wound on the inside of tube *n*, six turns to the inch in the middle and closer at the ends to partly compensate for radiation at the ends; *b*, alundum extraction thimble No. 5811, 30 mm. diameter, 80 mm. long; *j*, *i*, *g* and *h*, alundum disks; *o*, observation hole; *f*, Pt-PtRh thermo-element; *a*, ash cone.

FIG. 1.—Platinum Resistance Furnace.

Factors Influencing Softening Temperature.—The whole phenomenon of softening and fusion of ash is a result of chemical reaction, melting and solution, in which the time factor and fineness of division of the constituents are of the greatest importance. Some reactions between ash constituents, and of certain oxides with the atmosphere in which the ash is heated, take place

before any melting begins. Other reactions of various velocities make their appearance after incipient fusion. Also certain constituents have a slow time rate of melting, as, for example, silica and feldspar. These substances can be heated above their melting points and yet remain in a crystallin form for some time.

It is obvious, then, that the softening temperature of an ash cone must be affected by (1) the size, shape and inclination of

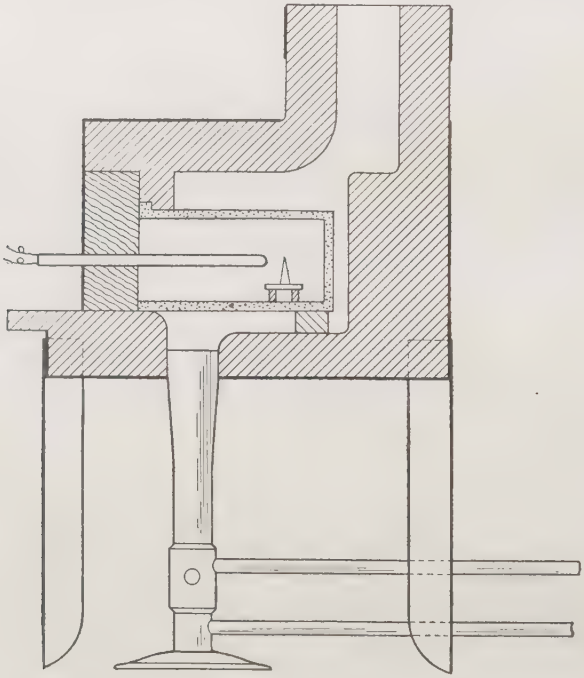


FIG. 2.—Meker Muffle Furnace, No. 29.

the cone; (2) the fineness of the ash; (3) the rate of heating; and (4) the nature of the atmosphere in which the cone is heated.

Influence of Iron Oxides.—Owing to the invariable presence of iron oxide in coal ash, the oxidizing or reducing nature of the surrounding gases determines the rôle which the iron shall play in the slagging reactions. In the ordinary method of preparing a coal ash by burning off the carbon in air at a red heat, the iron content is practically all converted to ferric oxide (Fe_2O_3), which

is stable in air at temperatures up to 1350° C.[1] At this temperature it dissociates into magnetite (Fe_3O_4) and oxygen. Therefore in determining the softening temperature of an ash in air, with absolutely no reducing gases present, we are dealing with the formation of slags in which the iron component enters the reaction as ferric oxide or magnetite. Such slags are more refractory[2] and more viscous[3] than those found by heating mixtures containing the iron component in the ferrous state.

We may therefore expect lower softening temperatures if the atmosphere surrounding the cone is able to reduce the ferric oxide to the ferrous state before fusion begins, or during the first stages of incipient melting. On the other hand, if the concentration of the reducing gases is so great that the iron oxide is reduced to metal, the softening temperature will again be increased, since one of the most active fluxing constituents, iron oxide, is removed from the system. Most ashes have already an excess of high-melting constituents like silica and alumina, so that any considerable reduction of iron oxide to metallic iron would tend to increase the refractory effect of the silica and alumina. Hence lower softening temperatures may be expected in tests made in gas furnaces where some reducing gases come in contact with the ash, while higher results should be found both in furnaces where air only surrounds the cone, and in carbon or graphite electric furnaces where strongly reducing atmospheres reduce the iron oxides to metallic iron.

EXPERIMENTAL.

Description of Furnaces.—In the following experiments the authors have studied the effect of various factors in the softening temperature of ash, by making tests on a selected series of coal ashes in different types of furnaces such as are in more or less common use for the purpose. Each of these furnaces has its own particular atmosphere (oxidizing, reducing, or neutral), which, as will be shown, is the principal cause of the large varia-

[1] Walden, "The Dissociation Pressure of Ferric Oxide," *Journal*, Am. Chem. Soc., Vol. 30, p. 1350 (1908).

[2] Peters, "Principles of Copper Smelting," Hill Publishing Co., New York, pp. 399–400, (1907).

[3] Greiner, "Ueber die Abhängigkeit der Viscosität in Silikat Schmelzen von ihrer Zusammensetzung," Inaugural Dissertation, Jena, p. 55 (1907).

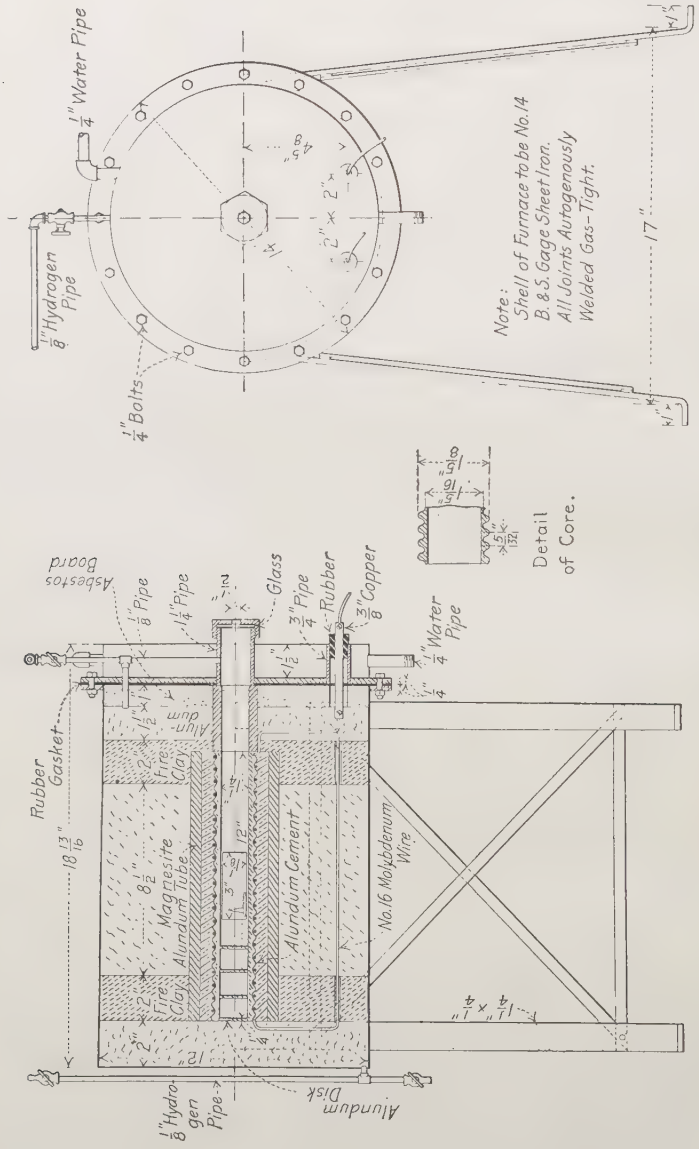
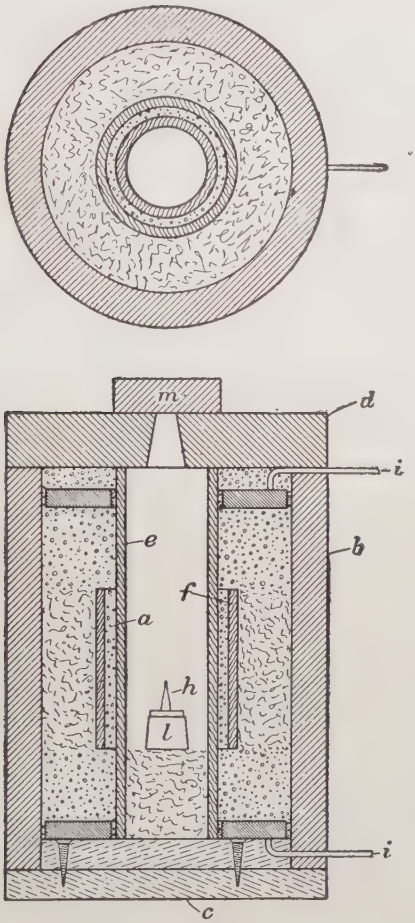


FIG. 3.—Molybdenum-Wire Resistance Furnace.



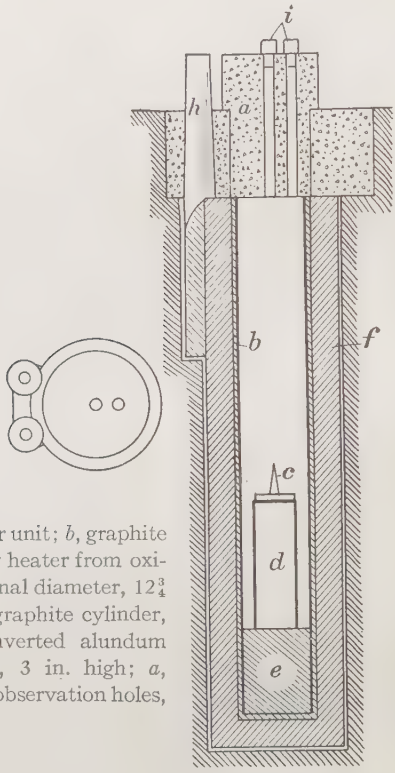
b, fireclay cylinder, 8 in. external diameter, 10 in. high, 1-in. wall; *d*, fireclay cover plate, 2 in. thick; *m*, fireclay plate covering peep-hole; *f*, alundum tube, 3-in. bore, 4 in. long, $\frac{5}{16}$-in. wall; *e*, alundum tube, 2-in. bore, 9 in. long, $\frac{1}{4}$-in. wall; *a*, granular carbon resistor consisting of electrode carbon crushed to pass a 6-mesh and remain on a 12-mesh screen; *i*, *i*, wrought-iron electrodes; *c*, granular carbon; *l*, inverted alundum crucible; *h*, ash cone.

FIG. 4.—Carbon Resistance Furnace.

tions that were found on comparing the softening temperatures of the same sample, in the different furnaces.

The following six furnaces were used in the comparative tests:

1. Platinum resistance furnace (Fig. 1), having an oxidizing atmosphere of air, with absolutely no reducing gases present.



f, graphite heater unit; *b*, graphite tube for protecting heater from oxidation, $1\frac{5}{8}$ in. internal diameter, $12\frac{3}{4}$ in. long; *c*, solid graphite cylinder, 2 in. high; *d*, inverted alundum extraction capsule, 3 in. high; *a*, refractory plug; *i*, observation holes, $\frac{1}{4}$ in. diameter.

FIG. 5.—Heater Unit of Northrup Furnace.

2. Meker muffle furnace No. 29 (Fig. 2), having a mixed atmosphere of air and combustion gases.

3. Muffle furnace No. 2, having a mixed atmosphere of air and combustion gases, from the flames surrounding the porous fire-clay muffle.

4. Molybdenum-wire resistance furnace (Fig. 3), having a reducing atmosphere of hydrogen.

5. Granular carbon resistance furnace (Fig. 4), having a reducing atmosphere of carbon monoxide.

6. Northrup graphite resistance furnace (Fig. 5), having a reducing atmosphere of carbon monoxide.

Before giving the results obtained in each furnace, the coals tested and the general method of operation will be described.

TABLE I.—DESCRIPTION AND ORIGIN OF SAMPLES.

Sample No.	Laboratory No.	Description.	Bed.	Location of Mine.		
				Nearest Town.	County.	State.
1	15840	Bituminous	Coal Creek	Fraterville	Anderson	Tenn.
2	15841	Bituminous	American	Parrish	Walker	Ala.
3	15842	Bituminous	Sterling	Manring	Claiborne	Tenn.
4	15843	Bituminous	Jellico	Jellico	Campbell	Tenn.
5	15844	Bituminous	Mingo	Fork Ridge	Claiborne	Tenn.
6	15845	Bituminous	Coal Creek	Oliver	Morgan	Tenn.
7	15846	Bituminous	Mary Lee	Red Star	Walker	Ala.
8	15847	Bituminous	Nickel Plate	Jefferson	Jefferson	Ala.
9	15848	Bituminous	Thompson	Marvel	Bibb	Ala.
10	16018	Bituminous	Red Stone	Lemely Jct	Barbour	W. Va.
11	16019	Bituminous	Pittsburgh	Morgantown	Monongalia	W. Va.
12	16243	Bituminous	No. 5	Booneville	Warrick	Ind.
13	16583	Semi-bituminous	Pocahontas No. 3	Simmons	Mercer	W. Va.
14	16584	Semi-bituminous	Pocahontas No. 3	Bramwell	Mercer	W. Va.
15	16585	Semi-bituminous	Pocahontas No. 3	Elk Ridge	McDowell	W. Va.
16	16586	Semi-bituminous	Pocahontas No. 3	Big Four	McDowell	W. Va.
17	16587	Semi-bituminous	Pocahontas No. 3	Jenkin Jones	McDowell	W. Va.
18	16589	Semi-bituminous	Pocahontas No. 3	Boissevain	Tazewell	W. Va.
19	17081	Anthracite (buckwheat)				Penna.

Description and Analyses of Samples.—The description and origin of the coal samples are given in Table I, and the analyses of the ash and partial analyses of the coal in Table II. As may be seen from an inspection of these tables, the series of fuels tested include anthracite, bituminous, and semi-bituminous coal; the composition of the ash covers a fairly wide range, silica varying from 12.3 to 56.0 per cent; alumina from 12.2 to 34.7; ferric oxide from 4.5 to 69.7; lime from 0.6 to 12.6; and magnesia from 0.2 to 1.9 per cent.

Preparation of Ash.—The coal samples were ground to 60-mesh with crusher, rolls and ball mill as described in Technical Paper No. 8.[1] The 60-mesh material was spread out on shallow, 6-in. fire-clay roasting dishes, and completely ashed with occasional stirring in a muffle furnace at a temperature not exceeding 750° C. All of the ash was finally put through a 100-mesh screen and thoroughly mixed.

Preparation of Cones.—Sufficient ash to make the desired number of cones was transferred to an agate mortar, moistened with 10-per-cent dextrin solution and worked into a plastic mass

TABLE II.—ANALYSES.

Sample No.	Analysis of Ash. Percentage of									Analysis of Dry Coal. Percentage of	
	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O	SO_3	Sulfur.	Ash.
1	35.7	23.5	32.9	1.2	3.2	1.1	0.3	1.1	0.5	3.1	10.8
2	47.3	34.6	9.8	1.8	1.3	0.4	2.1	2.5	0.1	1.7	17.4
3	55.8	33.5	5.0	0.9	1.5	0.7	0.5	2.2	0.1	0.8	15.6
4	43.3	31.4	13.6	1.6	4.2	1.4	0.6	2.9	1.4	1.5	8.6
5	42.2	30.6	19.0	1.2	1.3	1.0	1.3	2.9	0.2	1.4	7.5
6	12.3	12.2	69.7	0.4	3.9	0.7	0.3	0.6	0.2	5.8	8.2
7	54.1	34.7	4.5	1.5	1.2	0.9	0.6	2.5	0.2	0.7	17.7
8	46.8	28.7	18.0	1.4	0.6	0.9	1.6	2.4	0.3	2.3	12.7
9	54.8	27.0	7.0	1.3	4.3	1.7	0.3	3.1	1.4	0.6	17.5
10	38.4	24.2	22.4	1.1	7.7	0.9	0.3	1.9	3.8	2.1	7.9
11	50.4	24.0	20.4	1.4	1.7	0.2	1.0	1.0	0.3	2.7	10.9
12	37.1	17.6	35.9	0.7	3.2	0.9	0.4	1.8	2.3	5.8	11.5
13	54.8	27.0	7.8	1.6	1.6	1.5	2.2	1.9	0.5	0.7	8.0
14	54.8	29.2	6.9	1.8	1.4	0.6	1.9	2.1	1.0	0.7	7.5
15	54.1	24.8	9.4	2.3	4.0	1.4	1.0	0.8	2.8	0.6	7.1
16	37.2	25.5	11.8	1.5	12.6	1.9	1.4	0.4	5.6	0.6	5.9
17	51.1	25.2	10.1	1.8	5.1	1.6	0.8	0.9	3.1	0.6	6.8
18	51.8	25.0	9.0	2.0	4.0	1.5	1.3	0.8	4.3	0.7	5.6
19	56.1	31.4	5.0	1.9	1.0	1.0	0.7	3.1	0.5	0.7	21.5

with a spatula or pestle. After moistening the brass mold (Fig. 6) with kerosene to prevent sticking, the plastic material was firmly pressed into it with a knife spatula, and the surface struck off smooth to make a neat solid triangular pyramid. The cone was then pushed out of the mold by applying a small knife blade at the base. With a little practice and proper lubrication of the mold this can be done at once after molding, without waiting for the cone to dry.

After drying, the cones were mounted in a refractory base

[1] Stanton and Fieldner, "Methods of Analyzing Coal and Cokes," *Technical Paper No. 8*, Bureau of Mines, pp. 7–9 (1913).

made up of a mixture of two parts of kaolin to one part of alumina (Al_2O_3). This mixture was moistened with water to make it workable, enough taken for the base to be made and spread upon a sheet-iron plate. A small hole was made into which the cone was set, and the base material worked around the bottom of the cone so that it would be firmly set at the desired inclination in the base. The iron plate was then put on a hot-

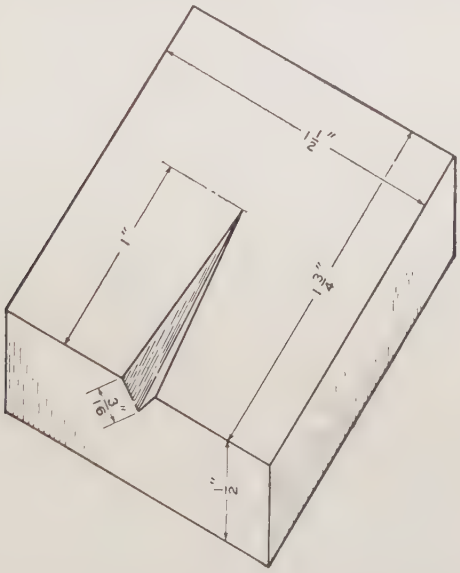


FIG. 6.—Brass Cone Mold.

plate and the mounted cones dried slowly until all water was driven off. The dextrin was then burned out by igniting the mounted cones in a muffle after which they were ready for use. In the earlier experiments the cones were made directly from the 100-mesh ash; later it was found that grinding the ash to an impalpable powder (or at least to pass 200-mesh) made a more substantial and more easily molded cone than 100-mesh material. Three different sizes of cones were used:

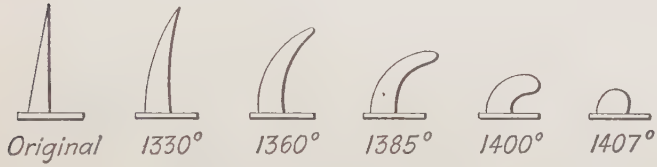
	SIDE OF BASE, IN.	HEIGHT, IN.
Cone No. 1	$\frac{1}{4}$	1
Cone No. 2	$\frac{1}{4}$	$1\frac{1}{2}$
Cone No. 3	$\frac{3}{16}$	1

General Method of Heating.—The general procedure in making a softening-temperature determination was the same with all the furnaces used, though necessarily the details varied in the different furnaces. The test specimen was put into the cold or nearly cold furnace, and the furnace heated at the rate of 10 to 15° C. per minute up to a point not less than 200° C. below the probable softening point, at which point the rate (usually 2 or 5° C. per minute) which had been adopted for that particular determination was begun. Temperature readings were taken every 5 minutes, and more frequently when approaching the softening temperature. Observations of the appearance of the cone were made at least as often as temperature readings were taken, special care being taken to note any deformation or warping due to shrinking before actual softening began. The point of initial softening or deformation was taken as the temperature where the first noticeable bending, rounding at the top, or swelling of the cone, took place. Warping of the cone due to shrinkage was not considered as beginning of fusion.

The softening point, deformation point, or so-called "fusion point," was taken as the temperature where the apex of the cone had bent over to touch the base, or failing to bend, had fused down to a lump or ball. Sketches (Fig. 7) were made of the appearance of the cone at the initial and final deformation points and at several intermediate points of deformation with the corresponding temperatures. Immediately after reading the temperature corresponding to complete deformation, the current or other source of heat was reduced so that the appearance of the cone could be verified by examination after removal from the furnace.

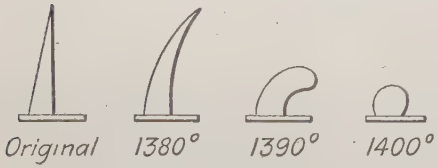
Temperature Measurements.—The temperature measurements were made in two ways, namely, by means of a Heraeus Pt-PtRh thermocouple with Siemens and Halske high-resistance millivoltmeter, in the platinum-resistance, Meker, and muffle No. 2 furnaces; and by means of a Wanner optical pyrometer in the molybdenum, carbon-resistance, and Northrup furnaces. The thermocouple and millivoltmeter were standardized from time to time in the physical laboratory of the Bureau under the direction of Dr. J. K. Clement by comparison with a standard thermocouple. The cold junction was kept at the temperature

Sample No. 16. Good Bend



Sample No. 15. Bent and Melted to a Ball.

Sample No. 5. Bent and Puffed.



Sample No. 11. Bent and Melted to a Ball.

FIG. 7.—Appearance of Ash Cones at Various Stages of Softening.

of melting ice during standardization and during use in measuring temperatures. Further checks on the system of furnace and pyrometer were obtained by placing crystals of pure diopside[1] in the positions occupied by the ash cones and noting the temperature of melting; usually the crystal melted between 1381 and 1395° C. (corrected temperature readings). The melting point of diopside is 1391° C.[2]

The Wanner pyrometer which was originally standardized by the Reichsanstalt, was rechecked by the Bureau of Standards after most of the work described in this paper was completed, and found to conform to the original standardization. It was checked daily against the amylacetate flame. As this pyrometer was used only with those furnaces which had strongly reducing atmospheres, it was possible to check the accuracy of the pyrometer readings and the black-body conditions of the furnace by placing thin strips of Kahlbaum's pure copper or Kahlbaum's pure nickel in place of the cones, and noting the apparent melting temperature. This was done at least once a week, and at the beginning and the end of each series of tests. The results obtained in these checks were usually within 10° C. of the true melting point at the copper point, and within 20° C. at the nickel point.

SUMMARY OF RESULTS OBTAINED IN THE DIFFERENT FURNACES.

Variations Due to Size and Shape of Cone.—Three different sizes of cones were compared in the Meker, the carbon-resistance, and the molybdenum furnace, namely, cone No. 1, $\frac{1}{4}$ by 1 in.; cone No. 2, $\frac{1}{4}$ by $1\frac{1}{2}$ in.; cone No. 3, $\frac{3}{16}$ by 1 in. The results which are given in Table III indicate that cones with a wider base as compared to height, as cone No. 1, give consistently higher softening temperatures than the more slender cones like cones Nos. 2 and 3. In general, the slender type of cone represented by size No. 3, $\frac{3}{16}$ by 1 in., was the most satisfactory in giving shorter and more definite softening intervals; it also gave less trouble from intumescence due to evolution of gases from the melting ash.

[1] Obtained through the kindness of Dr. Arthur L. Day, Director of the Geophysical Laboratory.

[2] Day and Sosman, "The Melting Points of Minerals in the Light of Recent Investigations on the Gas Thermometer," *American Journal of Science*, (4), Vol. 31, p. 346 (1911).

Effect of Inclination of Cone.—The effect of varying the position of the cone from vertical to an inclination of 45 deg. was quite appreciable, giving in some cases a lowering of 64° C. in the softening point (Table IV). A slight inclination from the vertical, not exceeding 25 deg., gave the same result as a vertical cone. The most satisfactory indication was obtained by mounting the cone with one side vertical, which usually caused the cone to bend towards that side. An important objection to mounting the cone at any considerable inclination, as 35 or 45 deg.,

TABLE III.—INFLUENCE OF SIZE OF CONE ON SOFTENING TEMPERATURE.

RATE OF HEATING, 2° C. PER MINUTE; 100-MESH ASH; CONES INCLINED 35 DEG. FROM VERTICAL; VALUES IN DEG. CENT.

Sample No.	Meker Furnace.			Carbon Furnace.			Molybdenum Furnace.		
	Cone No. 1.	Cone No. 2.	Difference.	Cone No. 1.	Cone No. 2.	Difference.	Cone No. 1.	Cone No. 2.	Difference.
13	1406	1273	+133	1390	1322	+68	1217	1217	0
15	1307	1186	+121	1311	1250	+61	1167	1161	+ 6
16	1225	1212	+ 13	1394	1341	+53	1241	1241	0
17	1242	1182	+ 60	1251	1220	+31	1192	1179	+13
18	1289	1204	+ 85	1316	1250	+66	1199	1185	+14
4	1265	1305	− 40	1357	1306	+51	1227	1234	− 7
5	1388	1376	+12	1401	1356	+45	1234	1231	+ 3
8	1357	1343	+ 14	1192	1166	+26	1318	1263	+55
9	1335	1318	+ 17	1327	1249	+78	1213	1199	+14
1	....	....	...	1100	1090	+10	1123	1123	0
2	....	....	...	1631	1593	+38	1430	1417	+13
3	....	....	...	1558	1562	− 4	1457	1396	+61
6	....	....	...	1207	1179	+28	1350	1336	+14
7	....	....	...	1663	1638	+25	1590	1580	+10
10	....	....	...	1187	1167	+20	1185	1192	− 7
11	....	....	...	1190	1139	+51	1220	1220	0
12	....	....	...	1088	1088	0	1068	1060	+ 8
14	....	....	...	1520	1424	+96	1326	1316	+10
Aver....	....	....	+ 46	....	....	+41	....	....	+11

is that certain ashes shrink before they reach their softening temperature, which allows the cone to fall over to a still larger inclination and thus indicate a premature down point.

Effect of Dextrin Binder.—The results of a few experiments made to show the effect of not burning out the dextrin binder before placing the ash cone in the molybdenum furnace are given in Table V. The maximum apparent effect was a lowering of 11° C. in the softening point. Mixing 10 per cent of dry dextrin with the ash caused a reduction of only 14° C. However, the conclusion that dextrin causes no material effect would only

TABLE IV.—EFFECT OF INCLINATION OF CONES.

TESTS MADE IN MOLYBDENUM FURNACE NO. 2; REDUCING ATMOSPHERE OF HYDROGEN; $\frac{3}{16}$ BY 1-IN. CONE; ASH GROUND TO AN IMPALPABLE POWDER; RATE OF HEATING 2° C. PER MINUTE; VALUES IN DEG. CENT.

Ash No.	Softening Point.			Difference.	
	Inclined 45 deg. from Vertical.	Inclined 25 deg. from Vertical.	Vertical Position.	Inclined 45 deg. and Vertical.	Inclined 25 deg. and Vertical.
39	1287	1283	1283	+ 4	0
42	1295	1313	1304	− 9	+9
9	1248	1304	1313	−65	−9
33	1396	1390	1390	+ 6	0
8	1414	1481	1478	−64	+3
19	1494	1510	1502	− 8	+8
2	1520	1520	1520	0	0
Aver...	....	....	....	−19	+2

TABLE V.—EFFECT OF DEXTRIN BINDER.

TESTS MADE IN MOLYBDENUM FURNACE NO. 2; ATMOSPHERE OF HYDROGEN; $\frac{3}{16}$ BY 1-IN. CONE, INCLINED 35 DEG. FROM VERTICAL; 100-MESH ASH; RATE OF HEATING 2° C. PER MINUTE; VALUES IN DEG. CENT.

Ash No	Softening Point.		Difference.
	Not Ignited.	Ignited.	
2	1492	1502	−10
8	1435	1434	+ 1
12	1260	1271	−11
8	1430	1434[a]	− 4
8	1427[b]	....	....
8	1420[c]	....	....
Aver.....		....	− 6

[a] Cone made in usual manner, then ignited in air at 850° C.
[b] One per cent dry dextrin mixed with the ash.
[c] Ten per cent dry dextrin mixed with the ash.

TABLE VI.—EFFECT OF FINENESS OF ASH.

TESTS MADE IN MOLYBDENUM FURNACE NO. 2; ATMOSPHERE OF HYDROGEN; $\frac{3}{16}$ BY 1-IN. CONE, PLACED IN A VERTICAL POSITION; RATE OF HEATING 2° C. PER MINUTE; VALUES IN DEG. CENT.

Ash No.	Softening Point.		Difference.
	100-mesh.	Impalpable Powder.	
39	1291	1283	+ 8
9	1248	1304	−56
42	1340	1313	+27
48	1380	1390	−10
8	1435	1481	−46
19	1519	1510	+ 9
2	1492	1520	−28
64	1108	1101	+ 7
65	1155	1127	+28
66	1174	1173	+ 1
67	1223	1185	+38
68	1216	1205	+11
69	1251	1237	+14
70	1259	1263	− 4
71	1327	1300	+27
72	1368	1350	+18
73	1380	1370	+10
Aver.	....	....	+ 3

TABLE VII.—COMPARISON OF SOFTENING TEMPERATURES OBTAINED IN MOLYBDENUM FURNACE, AT TWO DIFFERENT RATES OF HEATING.

ASH GROUND TO AN IMPALPABLE POWDER; $\frac{3}{16}$ BY 1-IN. CONE PLACED IN A VERTICAL POSITION; VALUES IN DEG. CENT.

Ash No.	Softening Point.		Difference.
	2° C. per Minute.[1]	5 and 2° C. per Minute.[2]	
58	1199	1220	−21
50	1220	1217	+ 3
49	1520	1514	+ 6
55	1380	1370	+10
28	1390	1394	− 4
52	1448	1478	−30
61	1365	1370	− 5
Aver.	....	....	− 6

[1] Furnace temperature was increased at the rate of 2° C. per minute until cone was down.

[2] Furnace temperature was increased at the rate of 5° C. per minute until deformation began; the rate was then reduced to 2° C. per minute until the cone was down.

apply under the condition of determining the softening point in hydrogen as was done in the above cited experiments.

Effect of Fineness of Ash.—Ash ground to an impalpable powder tended to soften at a slightly lower temperature than 100-mesh ash. As shown in Table VI, the difference was 3° C. and in no case exceeded 56° C. The finely pulverized ash could be molded into more substantial cones than the 100-mesh material.

Effect of Rate of Heating.—As stated in the introductory discussion, the rate of heating has an important bearing on the softening points indicated by silicate mixtures made in the form

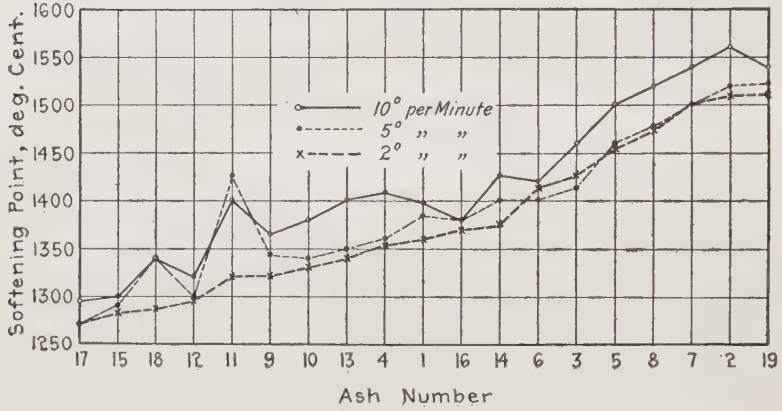


FIG. 8.—Effect of the Rate of Heating on the Softening Temperature in an Atmosphere of Hydrogen.

of Seger cones. The fusion reactions of such mixtures require time to approach equilibrium. Therefore higher results will be obtained with rapid heating. It is also probable that the reaction velocities will be different in oxidizing and reducing atmospheres. Tests were therefore made at rates of heating of 10, 5 and 2° C. per minute in an oxidizing atmosphere of air in the platinum furnace, and in a reducing atmosphere of hydrogen in the molybdenum furnace. The softening points obtained are given in Figs. 8 and 9. An inspection of these results shows that varying the rate of heating from 2 to 10° C. per minute causes much less increase of softening temperature in oxidizing atmospheres

than in reducing atmospheres of hydrogen. This would be expected because of the fact that the melting reactions in hydrogen are further complicated by reducing reactions, all of which are related to the time factor.

It is of course desirable to economize time by heating the test specimen as rapidly as possible consistent with avoiding unduly high results. This safe maximum rate of heating seems to be 10° C. per minute in purely oxidizing atmospheres.. However, in reducing atmospheres even 5° C. per minute gave in several cases results that were 50° C. higher than at 2° C. per minute. Further tests were therefore made in hydrogen, using a combined 5 and 2° C. rate of heating. The furnace temperature was increased at the rate of 5° C. per minute until deforma-

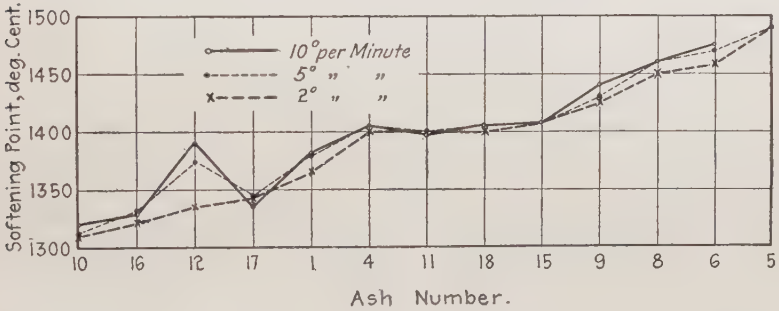


FIG. 9.—Effect of the Rate of Heating on the Softening Temperature in Air.

tion began. The rate was then reduced to 2° C. per minute until the cone was down. As shown by Table VII, the results approximate those obtained with the usual rate of 2° C.

Influence of Various Atmospheres.—None of the various factors affecting softening temperatures that have thus far been discussed seem capable of causing the exceedingly large differences in softening temperature that have been reported from different laboratories on the same samples of ash. For instance, Marks[1] has called attention to differences as great as 390° C. which were obtained on the same sample of ash by two different commercial laboratories. He himself obtained differences of

[1] "The Clinkering of Coal," *Journal*, Am. Soc. Mech. Engrs., Vol. 37, pp. 205-214 (1915).

200° C. on some duplicate samples which were tested in a Meker muffle furnace and a Hoskins graphite electric furnace. As stated by Marks, these large differences were undoubtedly due to the oxidizing or reducing action of the respective furnace atmospheres. It was realized at the outset of the investigation described in this paper that the reaction of the furnace gases with the iron constituent of the ash would largely determine the softening temperature. Therefore, comparative tests were made in the six different furnaces previously described, in order to show the effect of various oxidizing and reducing atmospheres.

The first series of oxidizing-atmosphere tests was made in an atmosphere of air in a platinum resistance furnace (Fig. 1),

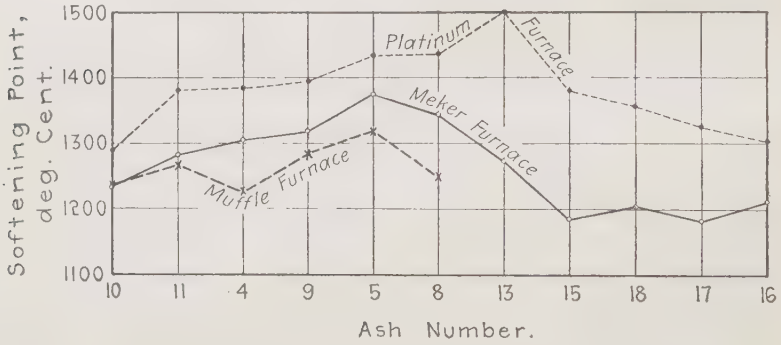


FIG. 10.—Comparison of Softening Temperatures Obtained in Meker Furnace, Muffle Furnace No. 2 and Platinum Furnace.

in which there could be no trace of reducing gases. The second series of tests was made in a gas-heated Meker muffle furnace (Fig. 2), and the third series in a fire-clay gas muffle furnace. As shown in Fig. 10, the platinum-furnace results are in all cases higher than those obtained in the gas furnaces. There was evidence of reduction by products of incomplete combustion from the fuel gases surrounding the muffles. The reduction was apparently greater in the muffle furnace than in the Meker furnace. This was probably due to the fact that the muffle furnace contained a rather porous fire-clay muffle, while the Meker furnace contained a special refractory muffle of dense texture.

From these results it is evident that softening-temperature tests made in gas furnaces cannot be referred to any standard atmosphere because of the uncertainty as to the relative concentration of the combustion gases that may penetrate the walls of the muffle. It is possible, perhaps, to maintain a sufficiently strong current of air through the muffle to render this concentration negligible; however, a much better method for softening tests in air is to use an electrically heated platinum-wire resistance furnace, in which there can be no question of possible reducing gases.

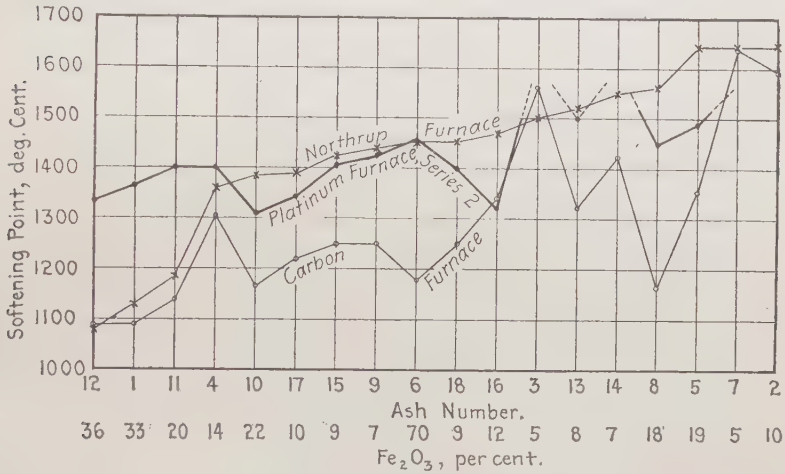


FIG. 11.—Comparison of Softening Temperatures Obtained in Northrup, Carbon Resistance and Platinum Furnaces.

In the tests made in the gas furnaces there was evidence of some reduction of ferric iron to the ferrous form, but not to metallic iron. To show the effect of strongly reducing atmospheres such as would cause the formation of metallic iron, tests were made in a granular carbon resistance furnace (Fig. 4) and a Northrup graphite furnace (Fig. 5), both having atmospheres of carbon monoxide (CO), and in a molybdenum resistance furnace (Fig. 3) having an atmosphere of hydrogen. The results obtained in the carbon and Northrup furnaces along with the platinum-furnace results are given in Fig. 11. As would be expected, the strongly reducing atmosphere of carbon

monoxide completely changes the softening characteristics of the ash, so that results widely different from those found in the platinum furnace in air, were obtained.

Since both the Northrup and granular-carbon resistance furnace produced atmospheres of carbon monoxide, similar softening temperatures were expected in the two series; that such did not prove to be the case is shown in Fig. 11. In 16 of 18 samples tested, the softening points in the Northrup furnace were higher than in the carbon furnace; the maximum was 396° C. and the average difference for the series was 134° C. The checks made on materials of known melting points, at various

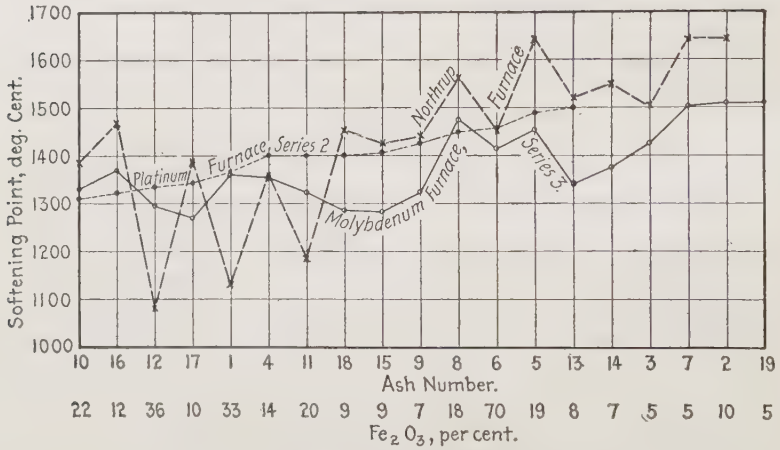

Softening Point, deg. Cent.
1700
1600
1500
1400
1300
1200
1100
1000
Platinum Furnace, Series 2
Northrup Furnace, Series 3.
Molybdenum Furnace,
10 16 12 17 1 4 11 18 15 9 8 6 5 13 14 3 7 2 19
Ash Number.
22 12 36 10 33 14 20 9 9 7 18 70 19 8 7 5 5 10 5
Fe2 O3, per cent.

FIG. 12.—Comparison of Softening Temperatures Obtained in Northrup, Platinum and Molybdenum Furnaces.

times during the course of these experiments, effectually rule out the possibility of attributing these large differences to errors of temperature measurement. The only explanation we have to offer is that in the carbon furnace the reduction of ferric oxide to metallic iron did not proceed as rapidly or as completely as in the Northrup furnace; in the latter furnace the ash cone is heated in a closed tube of graphite which excludes practically all air circulation, so that only nitrogen, carbon monoxide (CO), and a fractional percentage of carbon dioxide (CO_2) would surround the ash at temperatures above 1100° C. Such an atmosphere would reduce the ferric oxide to metallic iron and thus prevent

the formation of a low-melting ferrous silicate eutectic. On the other hand, the carbon furnace had an alundum tube between the carbon resistor and the cone, and it also permitted air to leak into the heating space as shown by the appearance of carbon-monoxide flames at the peep-hole and around the cover-plate. Sufficient carbon dioxide may therefore have been present to retard the reduction to metallic iron, thus leaving enough of the strongly fluxing ferrous oxide in the ash to form a low-melting fluid eutectic which caused the cone to collapse at a compara-

TABLE VIII.—COMPARISON OF SOFTENING TEMPERATURES OBTAINED IN DIFFERENT FURNACES, SHOWING EFFECT OF VARIOUS ATMOSPHERES.

RATE OF HEATING, 2° C. PER MINUTE; VALUES IN DEG. CENT.

Ash No.	Softening Points. Northrup Furnace,[2] CO+N	Carbon Furnace,[1] CO+CO_2+N	Molybdenum Furnace,[1] H	Muffle Furnace;[1] Air+Combustion Gases.	Meker Furnace;[1] Air+Combustion Gases.	Platinum Furnace,[2] Air.	Maximum Difference.
1	1131	1090	1360	1192	....	1365	275
2	1645	1593	1511	....	....	Above 1500	134
3	1502	1562	1427	....	....	Above 1500	135
4	1360	1306	1355	1226	1305	1400	174
5	1645	1356	1455	1320	1376	1490	325
6	1455	1179	1414	1318	....	1458	279
7	1645	1638	1502	....	....	Above 1500	143
8	1562	1166	1474	1251	1343	1450	396
9	1440	1249	1322	1284	1318	1425	191
10	1385	1167	1331	1199	....	1310	218
11	1185	1139	1322	1214	....	1400	261
12	1080	1088	1295	1182	....	1335	255
13	1520	1322	1340	....	1273	1500	247
14	1551	1424	1375	....	1417	Above 1500	176
15	1427	1250	1283	....	1186	1407	241
16	1470	1341	1370	....	1212	1322	258
17	1390	1220	1271	....	1182	1343	208
18	1455	1250	1287	....	1204	1400	251

[1] Ash 100-mesh; ½ by 1½-in. cone inclined 35 deg. from vertical.

[2] Ash ground to an impalpable powder; $\frac{3}{16}$ by 1-in. cone placed in vertical position.

tively low temperature. Examination of polished sections of fused cones from both furnaces disclosed more metallic iron in those from the Northrup furnace.

The results obtained with the series tested in the hydrogen atmosphere of the molybdenum furnace are compared with the platinum and Northrup furnace series in Fig. 12. It is quite evident that the softening temperature in hydrogen is again different from that in air or carbon monoxide.

A comparison of the softening temperatures obtained in the

six different furnaces is given in Table VIII. The maximum differences range from 134 to 396° C. In 14 samples which softened below 1500° C. in the platinum furnace in air, 9 gave their highest results in the strongly reducing carbon-monoxide atmosphere of the Northrup furnace (see Fig. 11); and 5 gave their highest results in the oxidizing atmosphere of the platinum furnace. The lowest softening points in 14 samples were distributed among the different furnaces as follows: Carbon furnace, 6; Meker furnace, 5; muffle furnace, 2; Northrup furnace, 1.

There seems to be no consistent relation between the results obtained in the different furnace atmospheres. Some ashes have a higher softening point in an oxidizing atmosphere, others in a reducing atmosphere of carbon monoxide. The only general conclusion that can be made regarding the effect of the atmosphere is that a given ash will have its highest temperature of softening in either an atmosphere of air, free from all traces of reducing gases, or in a strongly reducing atmosphere in which the concentration of reducing gases is so great that the iron constituents of the ash are mostly reduced to the metallic form. Between these two extremes of oxidation and reduction, there seems to be a condition of slight reduction by mixed gases such as was found in the muffle furnaces, in which reduction of ferric oxide proceeds mainly to the ferrous state. On account of its strongly fluxing action ferrous oxide greatly lowers the temperature of slag formation.

APPEARANCE OF CONES FUSED IN VARIOUS ATMOSPHERES.

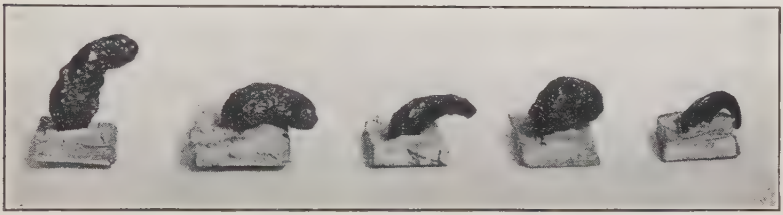
Platinum Furnace, Atmosphere of Air.—In Fig. 13 are shown the various ways in which cones of different ashes deform on heating in air in the platinum furnace. The manner of deformation is somewhat characteristic for each individual ash: No. 59 illustrates the typical Seger-cone bend; No. 26, the type which fuses to a lump; No. 29, the intumescing type; No. 7 is an ash which did not soften at 1500° C. The color of the fused cones varied from a buff, or reddish-brown, to a glossy black. The black cones were magnetic.

Molybdenum Furnace, Atmosphere of Hydrogen.—Fig. 14 illustrates typical forms from the molybdenum furnace (hydrogen

atmosphere). Owing to the reducing atmosphere, these cones almost always developed a pronounced vesicular structure. In some cases, as in ash No. 3, the swelling due to the evolution of gas, prevented the cone from bending in the normal manner. This is one of the reasons for the somewhat larger variations

7 29 26 1 59

FIG. 13.—Typical Forms of Cones Fused in Air in the Platinum Furnace.



3 19 17 14 10

FIG. 14.—Typical Forms of Cones Fused in Hydrogen in the Molybdenum Furnace.

4 15 8 17 11

FIG. 15.—Typical Forms of Cones Fused in Carbon Monoxide in the Northrup Furnace.

which occurred between duplicate determinations in reducing atmospheres. The fused cones were usually light to dark gray or grayish-black in color; they had a vitreous luster and invariably showed iron globules on examination under the microscope.

Northrup Furnace, Atmosphere of Carbon Monoxide and Nitrogen.—The cones fused in the Northrup furnace (CO atmosphere), all contained metallic iron, and varied from a dark gray to a brownish-black or metallic black color. The surface lacked the vitreous luster of the cones fused in air or hydrogen. Carbon was deposited on the cones and base in increasing quantity as the temperature exceeded 1400° C., as shown by the blackened base of ash No. 8 (Fig. 15) which was heated to 1550° C.

Another peculiarity in the Northrup series was the softening and swelling of the thick bottom portion of some of the cones before the apex began to soften. Ash No. 4 began swelling at the base at 1200° C.; heating was continued at the rate of 2° C. per minute until a temperature of 1375° C. was reached, when the current was interrupted and the furnace allowed to cool. As shown in Fig. 15, fusible constituents appeared to separate and run down the sides of the cone, leaving a refractory skeleton standing. However, on breaking the cone it was found that the irregular contour was due to the evolution and expansion of gases in the interior of the softening slag. The expanded bottom was a thin-shelled bubble of slag, as was the case in ashes Nos. 15, 8 and 17.

The first perceptible swelling of the lower part of ash No. 8 took place at 392° C. below the point where it finally bent over in the position shown in the photograph.

The earlier fusion of the thick part of the cone may be due to the inability of the carbon monoxide to penetrate and reduce the iron oxide in the interior, to metallic iron as rapidly as in the thin upper part. This phenomenon of irregular softening was noted only in the Northrup and carbon-resistance furnaces.

STANDARD METHODS FOR DETERMINING THE SOFTENING TEMPERATURE OF ASH.

Let us now consider the question of a standard method for determining the softening temperature of ash.

Obviously, the statement that an ash has a "fusing" or softening temperature of 1500° C. means nothing unless the exact conditions of making the test are defined. The method must therefore be carefully standardized in all its details before comparable results may be secured by different workers. The

most important consideration is the atmosphere in which the test shall be made. From the mere standpoint of securing results that can be duplicated in different laboratories, the softening temperature in air, as was determined in the platinum furnace, is undoubtedly the most satisfactory standard; but, unfortunately, ash-forming in a fuel bed is not free from the effect of reducing gases. Consequently, clinkering may take place under conditions of partial reduction similar to those which prevailed when the low softening points were obtained in the preceding experiments. Therefore, the danger point as regards clinker formation is the lowest possible softening temperature of an ash, under such conditions of reduction as are probable in a fuel bed.

The problem then resolves itself into an investigation of the effect of various mixtures of a reducing gas and its product of oxidation on the softening temperature with a view to finding that mixture which will give the lowest softening temperature. For experimental reasons we have chosen hydrogen as the reducing gas, since any desired mixture of hydrogen and water vapor can readily be secured by bubbling the hydrogen through water at the proper temperature.

EXPERIMENTAL METHOD.

Description of Furnace.—The arrangement of furnace and saturator is shown in Figs. 16 and 17. A Heraeus platinum-foil resistance furnace which happened to be available was equipped with an extra heater of 0.03-inch platinum wire, *h*, wound on the inside of the alundum tube, *e*, and connected in series with the platinum-foil[1] heating unit of the Heraeus furnace.

This alundum heater tube was supported on two alundum rings, leaving a $\frac{1}{2}$-in. air space between the heater tube and the Heraeus porcelain tube. Two additional alundum tubes, *c* and *u*, were placed similarly, one at each end of the platinum-wound tube. Through the furnace, modified in this way, was passed a tube, *AB*, of fused silica, glazed on both sides, $\frac{5}{8}$ in. inside diameter and 17 in. long.

The gas mixture entered the silica tube through the brass end-piece *B*, which was specially designed to make a gas-tight

[1] A nichrome heating element could as well be used for the outer heating unit.

connection by means of the brass bushing, *j*, and the asbestos packing, *n*. A removable glass window, *m*, permitted removal and insertion of cones in the furnace, and observation of deformation, and of temperature with the optical pyrometer. The glass was held in place between asbestos washers by a threaded brass ring.

An air bath *a* (Fig. 18), was placed around the end-piece *B*, to maintain its temperature above 100° C. and thus avoid any condensation of water vapor; the exit end *A* was made sufficiently short so that its temperature should be above 100° C. The gas mixture passed out through the glass capillary tube inserted in the rubber stopper at *A*.

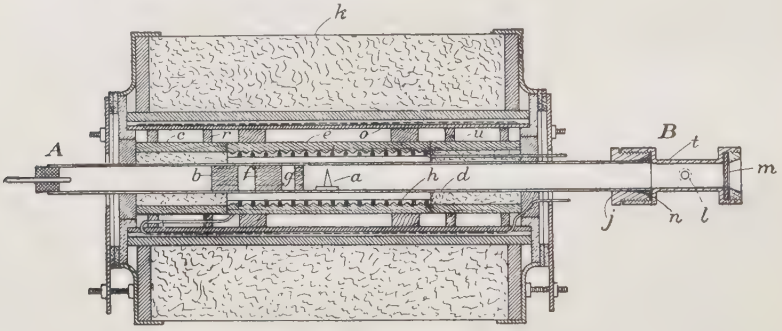


FIG. 16.—Vertical Longitudinal Section through Furnace.

Alundum stops, *b*, *f* and *g*, were placed at intervals back of the middle of the inner heater to provide a suitable background for taking readings with the optical pyrometer. These stops were made of thin-walled alundum tubes closed at one end, fitting snugly in the silica tube. To avoid rapid devitrification of the fused silica tube, temperatures were not carried above 1400° C.

Description of Saturator.—The apparatus by which various mixtures of hydrogen and water vapor were formed depends upon the saturation of pure hydrogen gas with water vapor at temperatures ranging from zero to within a degree or two of the boiling point. It is shown in Fig. 17 with furnace connections as used, except the hot-air bath surrounding the brass end piece, *m*, and side tube, *l*.

The saturator consists of a glass tube, *e*, $1\frac{3}{4}$ in. in diameter and 9 in. long, closed at the ends with rubber stoppers. Through the upper stopper, *h*, passes the thermometer, *d;* the outlet tube, *r*, communicating with the furnace through the brass tube, *l;* and a compact, adjustable vapor-pressure thermo-regulator, *c*, with electrical connections controlling through a relay the current in the heating coil, *f*. The thermo-regulator

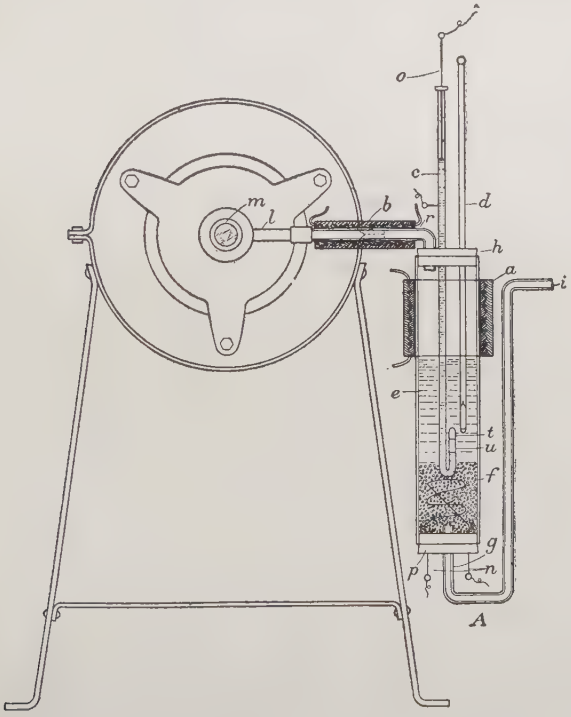


FIG. 17.—Front Elevation of Furnace and Vertical Section through Saturator.

is essentially that described by Feild,[1] without the refinement of being independent of atmospheric pressure. It consists of a bent-glass tube, in the short arm of which is a small quantity of liquid *u*,—ether, alcohol, or water, depending on the temperature desired,—resting on a mercury column, *c*. Above the liquid,

[1] "An Electrical Contact Vapor Pressure Thermo-regulator," *Journal*, Am. Chem. Soc., Vol. 36, pp. 72–76 (1914).

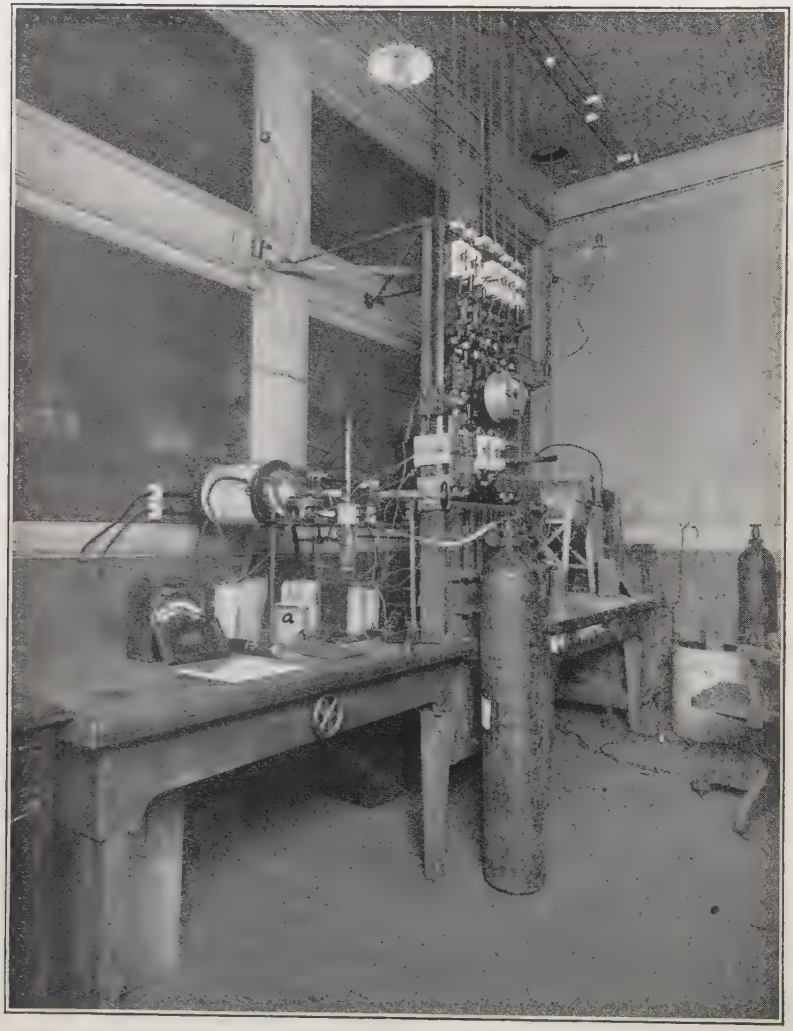


FIG. 18.—Arrangement of Furnace and Accessories.

u, is a small quantity of its saturated vapor, *t*, the pressure of which depends only upon the temperature and determines the height of the mercury column, *c*. This mercury column makes and breaks contact with the platinum wire, *o*. The temperature control, with a continuous stream of hydrogen bubbling around the glass beads, *f*, and through the water, *e*, was within 0.1 to 0°.2 C., which was sufficiently accurate for the purpose of the experiments. Hydrogen stored in a high-pressure cylinder was introduced at *i*, and its flow so adjusted as to give the desired current of hydrogen and water vapor through the fused-silica furnace tube. The water vapor present in the mixture of hydrogen and water vapor was prevented from condensing to liquid water on its passage to the furnace by two heating coils, *a* and *b*, constructed of nichrome wire wound on alundum cores and heated, similarly to the immersed coil, *f*, by a current from a low-voltage transformer operating at about 11 volts. An air bath of asbestos paper *a* (Fig. 18), heated by a small electric hot-plate, was placed around the brass end piece, *m*, and side tube, *l*.

Analyses were made of the gas and water-vapor mixtures issuing from the end of the silica furnace tube. With the saturator adjusted for a 50:50-per-cent mixture, 52 per cent hydrogen and 48 per cent water vapor were found; adjusting for an atmosphere of 89 per cent of water vapor, 91 per cent was found. This agreement was considered satisfactory.

Preparation of Test Specimen.—The ash, ground to 200 mesh, was molded with the aid of dextrin solution into a slender triangular pyramid or Seger cone and mounted with one side vertical, in a refractory base according to the method previously described. On account of the small diameter of the fused-silica tube, it was possible to use only the upper half of the standard $\frac{3}{16}$ by 1 in. cone which was adopted in the former experiments. The dextrin binder was burned off before placing the cone in the furnace, by ignition at a red heat in a muffle.

Measurement of Temperature.—The temperatures were read with a Wanner optical pyrometer, which was sighted on the thin alundum disk just back of the cone. A correction for absorption of the glass window was made.

The pyrometer was compared daily with the standard amylacetate flame, and once each week a thin piece of Kahl-

baum's pure copper foil was substituted for the ash cone and its melting point read in an atmosphere of hydrogen. The results were between 1075 and 1090° C. (melting point = 1083° C.).

EXPERIMENTAL RESULTS.

Softening-temperature determinations were made upon five different samples of coal ash (see Tables IX and X) containing from 7 to 69 per cent of ferric oxide (Fe_2O_3), in various mixtures ranging from pure dry hydrogen to pure water vapor. The

TABLE IX.—DESCRIPTION AND ORIGIN OF SAMPLES.

Laboratory No.	Description.	Bed.	Location of Mine.		
			Nearest Town.	County.	State.
15 848	Bituminous	Thompson	Marvel	Bibb	Ala.
16 586	Semi-bituminous	Pocahontas No. 3	Big Four	McDowell	W. Va.
15 844	Bituminous	Mingo	Fork Ridge	Claiborne	Tenn.
16 243	Bituminous	No. 5	Boonville	Warrick	Ind.
15 845	Bituminous	Coal Creek	Oliver	Morgan	Tenn.

TABLE X.—ANALYSIS OF ASH.

Laboratory No.	Percentage of								
	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O	SO_3
15 848	54.8	27.0	7.0	1.3	4.3	1.7	0.3	3.1	1.4
16 586	37.2	25.5	11.8	1.5	12.6	1.9	1.4	0.4	5.6
15 844	42.2	30.6	19.0	1.2	1.3	1.0	1.3	2.9	0.2
16 243	37.1	17.6	35.9	0.7	3.2	0.9	0.4	1.8	2.3
15 845	12.3	12.2	69.7	0.4	3.9	0.7	0.3	0.6	0.2

temperature was raised at the rate of 10 to 15° C. per minute to approximately 900° C., then 5° C. per minute until deformation began, and at 2° C. per minute thereafter until the cone was down. The dry hydrogen was obtained by bubbling the hydrogen through a wash bottle of concentrated sulfuric acid. The 100-per-cent water-vapor atmosphere was obtained by cutting off the hydrogen from the saturator and heating the water to boiling.

All the results obtained are given in Table XI, in which the softening temperature is given as a temperature interval expressed

by two values; the lower value before the hyphen is the temperature at which deformation (rounding of the apex or bending of the cone) began, and the higher value following the hyphen is the temperature at which the cone had bent over so that the

TABLE XI.—SOFTENING TEMPERATURES, IN VARIOUS MIXTURES OF HYDROGEN AND WATER VAPOR.

Laboratory No.....	15 848	16 586	15 844	16 243	15 845
Percentage of Fe_2O_3.....	7.0	11.8	19.0	35.9	69.7
Atmosphere, Percentage of H_2O	Softening Temperatures, deg. Cent.				
0.0	1322–1360	1390–1402	Above 1400	1304–1370	Above 1400
0.7	.....	.....	.....	1331–1355	.....
5.7	1279–1290	1313–1360	Above 1400	.....	Above 1400
6.3	.....	.....	.....	1248–1263	.....
7.6	.....	.....	.....	1220–1270	.....
10.7	.....	.....	[a]–1340	.....	.....
12.4	.....	1279–1313	1155–1271	.....	.....
15.8	.....	.....	.....	.....	1304–1330
18.1	.....	.....	.....	1104–1198	.....
19.9	.....	.....	1173–1271	.....	.....
29.6	.....	.....	.....	1056–1064	.....
31.2	1179–1271	1227–1251	1173–1263	.....	1227–1322
41.7	.....	.....	.....	1048–1062	.....
51.2	.....	.....	.....	1072–1078	.....
57.5	1220–1292	1220–1258	1173–1255	.....	1104–1322
77.2	.....	.....	.....	[a]1095	.....
83.2	.....	.....	.....	[a]1095	.....
83.9	1173–1295	1241–1249	1227–1263	.....	1206–1350
90.3	.....	1227–1251	1206–1295	[a]1095	.....
94.8	.....	.....	.....	1048–1080	.....
100.0	1350–1370	1313–1322	Above 1400	1304–1313	Above 1402

NOTE.—The first temperature is that at the beginning of deformation; the second, that at the end of deformation or "down" point of cone.

[a] Starting point not observed.

apex touched the base, or failing to bend, had softened to a lump. This latter final deformation point is meant when reference is made to the "fusion," "softening" or "deformation" point of an ash.

The final deformation points are plotted in Fig. 19, in which the abscissas represent the percentages of water vapor in the mixture and the ordinates the temperatures of final deformation. It will be noted that each curve has a well-defined minimum in the neighborhood of 30 to 70 per cent water vapor. This is a fortunate circumstance, as it enables the formulation of a practical method of determining the lowest softening temperature of an ash in a mixture of approximately equal parts of hydrogen and water vapor. Apparently the gas mixture need

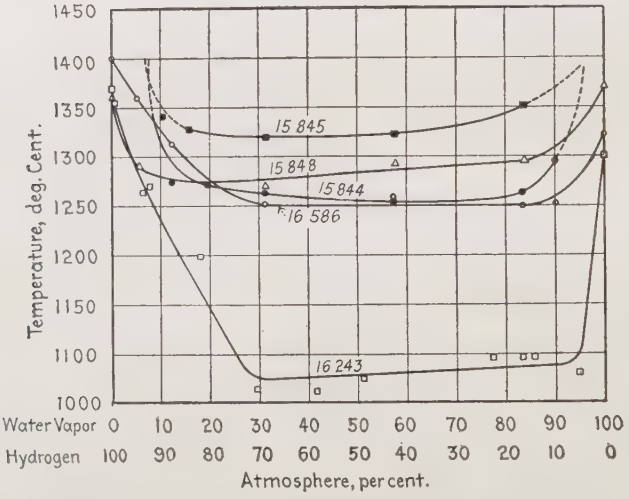


FIG. 19.—Effect of Various Mixtures of Hydrogen and Water Vapor on the Softening Temperatures of Coal Ash.

only be controlled between limits of 30 and 70 per cent of water vapor to insure the lowest softening temperature within the unavoidable experimental deviation in determinations of this character.

The sharp rise of the softening-point curves at both the oxidizing and reducing end confirms the reasons advanced in the foregoing part of this paper for the large variations in results obtained in different furnaces. It is evident that no concordant results can be obtained in oxidizing atmospheres containing small proportions of reducing gas, or similarly in essentially

reducing atmospheres with small admixtures of oxidizing gases, since in these regions the curves have a sharp inclination.

RELATION OF SOFTENING TEMPERATURE IN ATMOSPHERES OF HYDROGEN AND WATER VAPOR TO CLINKER FORMATION UNDER FURNACE CONDITIONS.

The general form of the softening-point curves in hydrogen and water vapor suggests a choice of three atmospheres under which uniform softening points may be determined, namely, (1) 100 per cent hydrogen; (2) 50 per cent hydrogen and 50 per cent water vapor; (3) 100 per cent water vapor or 100 per cent air, which gives nearly the same result as shown in Table XII.

TABLE XII.—COMPARISON OF SOFTENING POINTS OF SAMPLES IN AIR AND WATER VAPOR.

Laboratory No.	Softening Point, deg. Cent.	
	Air.[1]	Water Vapor.[2]
15 845	1458	Above 1400
15 844	1490	Above 1400
15 848	1425	1370
16 243	1335	1313
16 586	1322	1322

[1] Cone, $\frac{3}{16}$ by 1 in.
[2] Cone, $\frac{3}{32}$ by $\frac{1}{4}$ in.

The atmosphere to be selected for a standard softening-temperature test should be that one which produces a slag with the iron in the same state of oxidation as is usually found in fuel-bed clinkers. Determinations were therefore made of the relative percentages of ferrous, ferric and metallic iron in the glassy portions of furnace clinkers and in the ash cones fused in the three above-mentioned atmospheres.

FERROUS, FERRIC AND METALLIC IRON IN FUSED ASH CONES AND CLINKER SLAGS.

Twelve to 14 ash cones, aggregating about 2 g., were placed in a platinum boat, and heated in the silica tube of the furnace at the usual rate up to the softening temperature of the particular ash in the particular atmosphere in question. The boat and its

contents were held at this temperature for 30 minutes to allow the softening reactions to approach equilibrium. As in the case of softening-temperature determinations, the cones were heated to redness previous to the experiment to oxidize and remove the organic binder. At the end of the 30-min. heating period the electrical current was cut off and the furnace allowed to cool rapidly, without interrupting the gas stream. It would have been better in order to avoid any possibility of change during cooling to have quenched the fusion suddenly. However, the viscosity of these ash-melts was so great that little change was probable at the rate of cooling used. The fused material appeared glassy.

Fusions were made of sample No. 16,243 in 100 per cent hydrogen, 50:50-per cent mixture of hydrogen and water vapor, and 100 per cent water vapor; also of sample No. 15,848 in 50:50 hydrogen-water vapor, and sample No. 13,629 in air.

After cooling the furnace, the fusions were removed from the boat and were pulverized, first by impact in a diamond mortar of hardened steel, and then in an agate mortar, to 100 mesh, and analyzed for ferrous, ferric and metallic iron by the method of Mathesius.[1] The results given in Table XIII show well-defined differences in the state of oxidation of the iron content of the fusions in the various atmospheres. In hydrogen 78 per cent of the iron was reduced to metal; in steam and air approximately 70 per cent remained as ferric iron; in the 50:50-per cent hydrogen-water vapor mixture, 80 per cent or more was present as ferrous iron.

Together with these analyses of fused ash cones are given similar analyses of the fused and glassy portions of several clinkers produced under working conditions in the fuel beds of different boiler furnaces operating with Pittsburgh coal. The coal, however, came from different mines. Clinker No. 20,137 was obtained from the grate of a small hand-fired experimental furnace which was used for combustion investigations. Clinkers Nos. 20,145 and 20,452 were kindly furnished by Mr. A. A. Straub, Steam Engineer of the Duquesne Light Co. of Pittsburgh, Pa. These clinkers were dumped from the fuel bed while hot

[1] "Untersuchungen über die Reduzierbarkeit von Eisen erzen in Strömenden Gasen," *Stahl und Eisen*, Vol. 34, p. 866 (1914).

and were quenched with water. Both were of the particularly troublesome taffy-like variety that flow over the grate bars and shut off the air supply for combustion.

Fig. 20 is a photograph of clinker No. 20,452. The straight-line edge is the side which adhered to the corrugated grate of

TABLE XIII.—ANALYSES OF FUSED CONES OF ASH, AND OF GLASSY PORTIONS OF FURNACE CLINKERS.

Laboratory No.	Description.	Maximum Temperature of Fusion, deg. Cent.	Total Iron, per cent.	Percentage of Total Iron, Occurring as			Character of Slag.
				Metallic Iron.	Ferrous Iron.	Ferric Iron.	
15 848	Ash fused in atmosphere of 50 per cent hydrogen, 50 per cent water vapor.	1280	4.7	6	94	Trace	Light-gray color; non-magnetic.
16 243	Ash fused in atmosphere of 50 per cent hydrogen, 50 per cent water vapor.	1080	27.4	Trace	82	18	Glassy black; non-magnetic.
16 243	Ash fused in atmosphere of hydrogen.	1370	28.6	78	21	1	Black, metallic luster; contained particles of metallic iron, which were magnetic.
16 243	Ash fused in atmosphere of water vapor.	1300	27.9	Trace	28	72	Glassy black; surface had reddish tint; strongly magnetic.
13 629	Ash fused in atmosphere of air.	1400	15.5	Trace	30	70	Dark red to almost black; glassy; highly magnetic.
20 137	Clinker from a hand-fired furnace.	....	14.5	12	79	9	Dark glass; some pieces slightly magnetic.
20 145	Clinker from a boiler furnace equipped with Roney stoker, 20th St. station.	....	12.7	6	75	19	Brown to black glass; some pieces slightly magnetic.
20 452	Clinker from a boiler furnace equipped with Roney stoker, Brunot's Island station.	....	8.9	9	75	16	Black glass; slightly magnetic.

the new model, type D, Roney stoker. The matrix of this clinker was a black glass which could readily be chipped out in quantity for analysis without including any piece of coke or unfused material. At the contact surface of coke and slag, particles of metallic iron could be identified. A microscopic examination of the powdered slag from this clinker as well as

from the others, by Mr. A. A. Klein of the Bureau of Standards, showed the material to be essentially a glass with small quantities of sillimanite (Al_2O_3, SiO_2) and of an opaque iron mineral, probably magnetite, as the only crystallin phases present.

A comparison of the analyses of the fused ash cones, and of the three fuel-bed clinkers given in Table XIII, shows that of the different atmospheres employed in the laboratory furnace, the 50:50-per-cent hydrogen-water vapor mixture produced a slag which corresponded most closely in the state of oxidation of its iron content to the glassy portions of the furnace clinkers. In both cases approximately 80 per cent of the iron appeared as

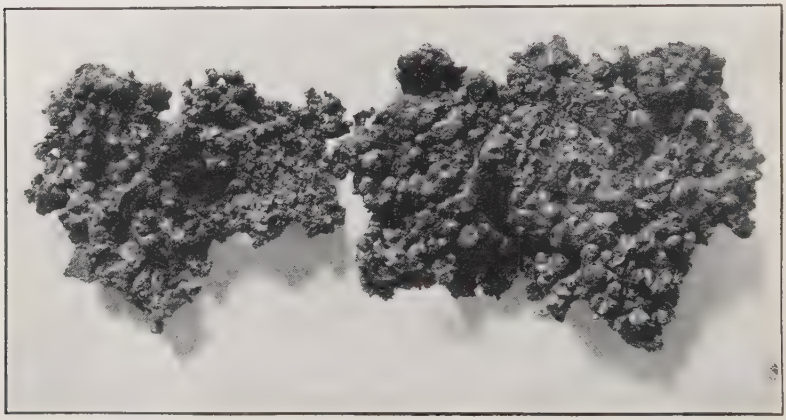


FIG. 20.—Clinker No. 20,452 from Grate of Roney Stoker.

ferrous iron, the form in which it imparts the maximum fluxing action on the silicate mixture.

We may therefore conclude that the minimum softening temperature of a coal ash as determined in a 50:50-per-cent atmosphere of hydrogen and water vapor, is more representative of the temperature of clinker formation under furnace conditions than the more or less higher results obtained in strongly reducing atmospheres of hydrogen, or carbon monoxide, on the one hand, and oxidizing atmospheres of air, water vapor or carbon dioxide on the other.

It should be understood in this connection that the recommendation of using a mixture of equal parts of hydrogen and

water vapor, as a standard atmosphere for determining the minimum softening temperature, is not based upon any supposition that this particular atmosphere is the one that prevails in a fuel bed. It is simply a convenient laboratory means of causing the iron component to be reduced to the ferrous state, in which it can exert its maximum fluxing action. In the fuel bed the reducing agents are solid carbon, carbon monoxide, hydrocarbon gases, etc.

In order to obtain direct evidence on the exact relation of this new minimum softening-temperature test to clinker formation under various fuel-bed conditions, the Bureau of Mines is now making practical clinker tests in a specially designed experimental furnace, in which different coals having ashes of various degrees of fusibility can be tested under similar and known conditions of combustion. In view of the fact that no softening-temperature test of the well-mixed sample of the average ash of a coal, takes into account the physical distribution of the impurities in the coal, much additional investigation on this point must be made before the exact relation of these laboratory fusibility tests to clinker formation can be established.

Summary and Conclusions.

The effect of various factors on the softening temperature of a number of typical coal ashes has been studied in eight different furnaces, having atmospheres of varying degrees of oxidation and reduction. The influences of the more important factors are summarized as follows:

1. *Size and Shape of Cone.*—Cones $\frac{1}{4}$ by $1\frac{1}{2}$ in. gave closer duplication and from 10 to 50° C. lower average softening points than $\frac{1}{4}$ by 1-in. cones; $\frac{3}{16}$ by 1-in. cones gave practically the same results as $\frac{1}{4}$ by $1\frac{1}{2}$-in. cones. The more slender cones were more satisfactory in giving shorter and more definite softening intervals; they also gave less trouble from intumescence due to evolution of gases from the melting ash. In reducing-atmosphere tests, the surrounding gases penetrated a thin cone more uniformly than one with a wide base.

2. *Fineness of Ash.*—Ash ground to an impalpable powder, tended to soften at a slightly lower temperature than 100-mesh ash. The difference averaged 3° C. and in no case exceeded 56° C.

Ash pulverized to at least 200 mesh could be molded into more substantial cones than 100-mesh material.

3. *Inclination of Cones.*—Mounting the cones with a considerable inclination, 35 or 45 deg. from the vertical, led in some cases to premature deformation points, which were caused by a further bending over due to shrinkage of the cone in its base, rather than deformation due to softening and flowing of the ash. Vertical or nearly vertical cones were free from this source of error, and gave the most concordant indications.

4. *Rate of Heating.*—In general, slower rates of heating gave lower softening points. Rates slower than 2° C. per minute are too time-consuming for practical consideration. Rates faster than 10° C. per minute led to inaccurate temperature measurements. Varying the rate of heating from 2 to 10° C. per minute caused less increase of softening temperature in oxidizing atmospheres than in reducing atmospheres of hydrogen (see Figs. 8 and 9). A 2° C. rate of heating gave the most uniform results; however, heating 5° C. per minute to initial deformation and then reducing to 2° C. per minute gave practically the same results and saved considerable time.

5. *Oxidizing or Reducing Atmosphere.*—The atmosphere in which the ash was heated proved by far the most important factor in causing large variations in the softening temperature. As pointed out in the introductory theoretical discussion, the highest softening points were obtained, either in an atmosphere of air (platinum furnace), or in a strongly reducing atmosphere of carbon monoxide (Northrup furnace) which prevented the iron oxide from acting as a fluxing agent by reducing it to metallic iron before the softening of the ash began.

The lowest softening temperatures were obtained in those atmospheres of mixed gases in which reduction of ferric oxide proceeded mainly to ferrous oxide, the most active phase of iron as regards slag formation at lower temperatures. The maximum variation in softening temperatures due to different atmospheres ranged from 134 to 396° C. (see Table VIII and Figs. 10, 11, and 12).

6. *Fusibility of Ash in Various Mixtures of Hydrogen and Water Vapor.*—Softening temperatures of five different coal ashes have been determined in various mixtures of hydrogen and

water vapor, ranging from 100 per cent hydrogen to 100 per cent water vapor. These results plotted in the form of curves showed that for each of the ashes tested there was a high softening temperature in pure hydrogen, on one end due to reduction of iron oxide to metallic iron; a similar high softening temperature in water vapor or air on the other end due to the iron oxide remaining for the most part in the form of ferric iron or magnetite; and a more or less lower softening temperature in the middle portion ranging from 30 to 70 per cent water vapor, due to the reduction of iron to the ferrous state in which it combined to form the readily fusible ferrous silicates.

7. *State of Oxidation of Iron in Clinker Slags.*—Analyses of actual clinker slags from two different boiler furnaces and one experimental furnace showed that fuel-bed conditions are such as to favor the formation of clinkers containing iron principally in the ferrous state.

8. *New Method of Determining the Minimum Softening Temperature of Ash.*—A new method of determining the minimum softening temperature of coal ash has been devised, in which the ash is heated in an atmosphere of approximately 50:50-per cent hydrogen and water vapor, whereby the iron oxide is caused to combine principally in the ferrous state, as actually found in fuel-bed clinkers.

ACKNOWLEDGMENTS.

The authors desire to express their deep appreciation of the assistance and of the many helpful suggestions received from Dr. G. A. Hulett, Chief Consulting Chemist of the Bureau of Mines, and from Mr. A. V. Bleininger, of the Pittsburgh station of the Bureau of Standards.

Some of the molybdenum-furnace tests were made by Mr. W. A. Mueller; the chemical analyses were made by Messrs. H. H. Hill, W. A. Selvig and F. D. Osgood, all of the chemical laboratory of the Bureau of Mines.

A number of the coal samples used in this investigation were furnished by Mr. E. G. Goodwin, Chief Fuel Inspector of the Southern Railway Co.

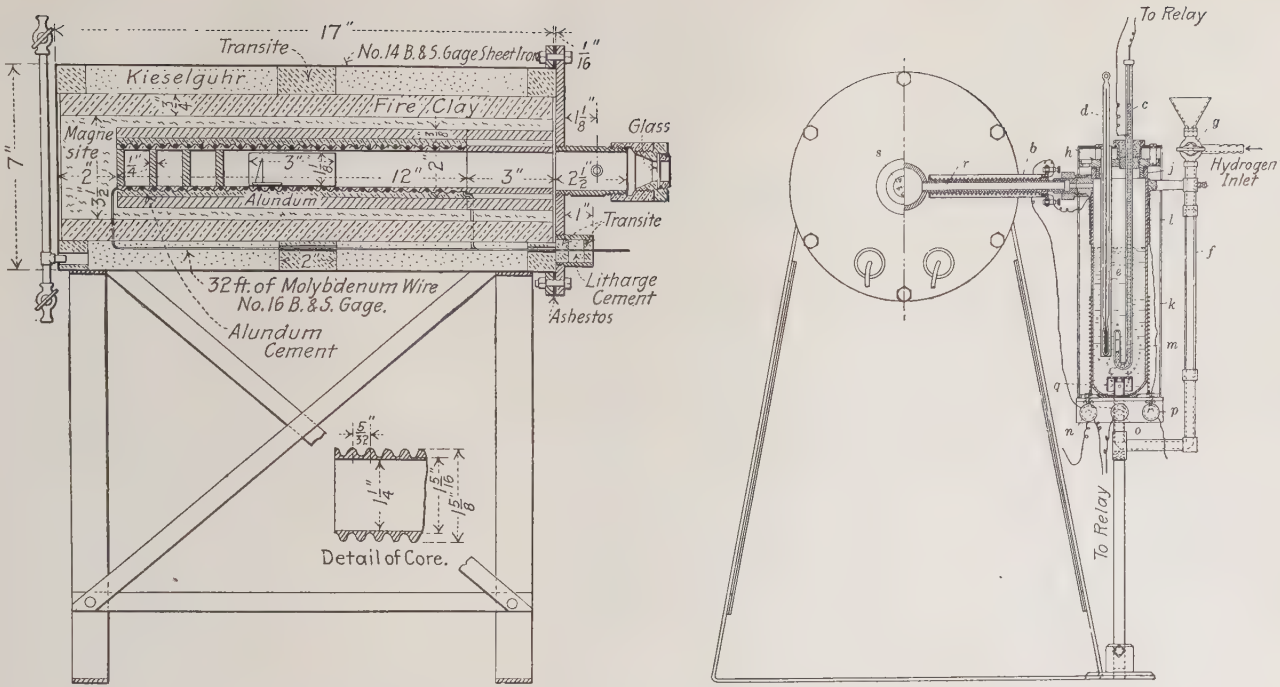
APPENDIX.[1]

The platinum-wire resistance furnace shown in Fig. 16, while satisfactory for the investigation of the effect of various atmospheres on the fusibility of coal ash, is not adapted to routine fusibility tests on account of the upper temperature limit of 1400° C. imposed by the use of a fused silica tube inside the wire-wound heater. Since writing this paper, a new form

FIG. 21.—Assembled Furnace and Saturator.

of molybdenum-wire furnace has been designed in which a temperature sufficiently high to fuse any coal ash can be

[1] This appendix was added to the paper after its presentation at the annual meeting.—ED.



(*a*) Furnace. (*b*) Saturator.

FIG. 22.—New Form of Molybdenum Furnace, with Saturator.

obtained, the upper limit being about 1700° C., the softening temperature of the alundum tube on which the wire is wound.

A photograph of the apparatus is shown in Fig. 21. A section of the furnace is shown in Fig. 22 (*a*). Instead of passing the gas mixture of hydrogen and water vapor through a fused silica tube, the furnace is made with a gas-tight, autogenously welded sheet-iron casing, so that the desired gas mixture can be maintained therein. The heating element is molybdenum wire, which is not oxidized in a 50 : 50 mixture of hydrogen and water

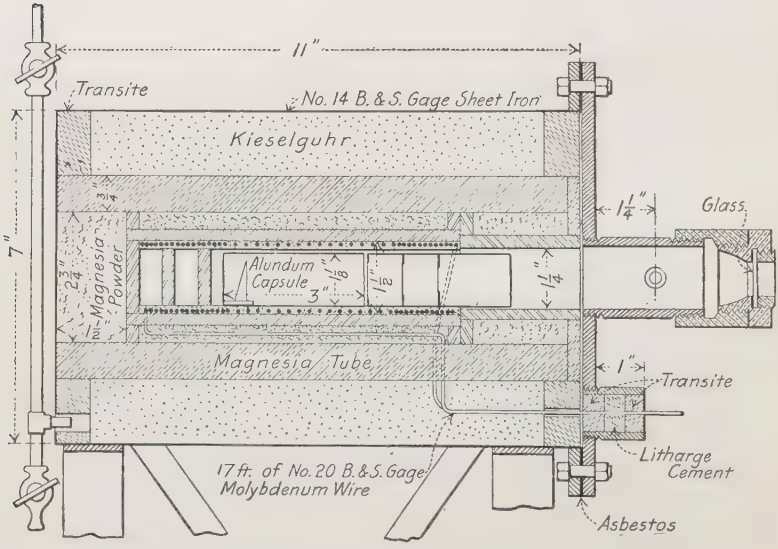


FIG. 23.—Vertical Longitudinal Section through Furnace.

vapor. The saturator shown in Fig. 22 (*b*) has also been modified and improved by making it of brass, thus doing away with the somewhat troublesome glass and rubber connections used in the experimental apparatus.

This apparatus has been used without requiring any repairs for a period of six months. It can be operated on a 110-volt A.C. or D.C. circuit, using an ordinary water rheostat for regulation; or on an A.C. line with a regulating transformer.

Fig. 23 illustrates a smaller modification of this new furnace,

which requires much less molybdenum wire, thus materially reducing the cost. Preliminary tests have given good results, and it will certainly be superior to the larger form if the smaller-caliber wire proves as durable as the larger size. At present it has not been used a sufficient length of time to determine its life in actual use.

DISCUSSION.

The President. PRESIDENT A. W. GIBBS.—I should like to ask whether any mechanical disturbances seem to start fusibility at a lower point. The reason I ask that question is that some coals, when allowed to burn quietly, do not seem to form much clinker, yet if disturbed, they form very large clinkers. I also wish to ask whether in making these determinations any mechanical disturbance, such as jarring, would modify the temperature reading at which fusion occurs.

Mr. Fieldner. MR. A. C. FIELDNER.—I think it is a well-recognized fact that clinker troubles are vastly increased when the fuel bed is subjected to frequent stirring; careless slicing or raking of a fuel bed is apt to mix the ash with the burning coal where it is subjected to a more reducing atmosphere, which increases the tendency to form the more fusible ferro-silicate slags. Stirring also brings the ash into a hotter zone of the fuel bed and tends to agglomerate small pieces of incipient clinker, which if left alone would not become particularly troublesome. Jarring would probably cause the cone to deform more rapidly and therefore give somewhat lower temperature readings.

Mr. Moldenke. MR. RICHARD MOLDENKE.—As an interesting comment on the question of fusibility of ash, I remember running across instances of what foundrymen thought a serious matter with their coke. It seems that some cokes, when put into shop salamanders for heating purposes, particularly when the column of incandescent coke is high, show drops of slag falling to the ground. Evidently the ash of these cokes is very fusible, and sufficient temperature is attained by natural draft up the salamander to melt it. Instead of being a detriment in cupola melting practice, this is rather an advantage and should be understood as such by foundrymen generally.

Mr. Camp. MR. A. D. CAMP.—I should like to ask Mr. Fieldner if he has ever made any experiments which show the effect of these atmospheres upon the quantitative determinations of ash in carbonaceous materials. Will the determination of ash in a

sample of coke or coal, performed in the reducing atmosphere of a gas-fired clay muffle, differ much from the result obtained by igniting the sample in the strongly oxidizing atmosphere of a platinum resistance furnace? Mr. Camp.

Mr. Fieldner.—Yes, we do get a difference in the weight of ash obtained in a muffle, due to oxidizing or partly reducing atmosphere. In an oxidizing atmosphere the highest product of oxidation, ferric oxide (Fe_2O_3), is obtained. In a partly reducing atmosphere this oxidation may be incomplete. There are also further differences in weight of ash obtained under various conditions due to reaction between pyrite (FeS_2) and calcium carbonate ($CaCO_3$) which are frequently present in coal. If no alkali or alkaline earths are present, the pyrite is completely oxidized to Fe_2O_3, the sulfur escaping principally as sulfur dioxide (SO_2). If calcium carbonate is present, it is more or less completely decomposed to CaO and CO_2, the CaO in turn being more or less converted to $CaSO_4$, which causes a gain in weight of the ash due to the fixation of a part of the sulfur that ordinarily escapes as a gas in the absence of lime. Mr. Fieldner.

Mr. H. S. Spackman.—This is a question which interests me from the fact that I have been struggling in a plant, with which I am connected, with one of the most aggravated cases of trouble from fusion of the ash I have ever heard of. The plant consists of five 400-h.p. Rust boilers, fired with Roney stokers. The peculiar part of this difficulty was that the ash fused on the tubes of the boilers. It was impossible to keep the boilers in constant operation over five days. At the end of that time the entire opening of the fire arch was closed up, and the space between the first three rows of tubes was filled with a solid mass of glassy slag, which would build up to the thickness of 4 in. on the front row of tubes. The fire was an oxidizing one, the gas analyses showing about 50 per cent excess air, and the temperatures were not extraordinary, ranging, as determined with an optical thermometer, from 2600 to 2700° F. in the hottest parts of the furnace. Mr. Spackman.

We have overcome the difficulty by purchasing another coal, which, however, is very high priced. We have been considering buying our coal under specifications as to the fusing point of the ash, but have encountered the problem of our

Mr. Spackman. inability to get agreement between the laboratory tests as to the fusing point of the ash and the fusing of the ash in the furnace. This question is a most important one, and deserving of a great deal of thought.

Mr. Hasse. MR. O. C. HASSE.—I should like to ask the author if he has any suggestions to make as to how to overcome the clinkering trouble in actual practice.

Mr. Fieldner. MR. FIELDNER.—Much clinker trouble can be avoided by proper methods of firing. Carry a thin fuel bed, keeping it as oxidizing as possible by taking the air necessary for combustion through the fuel bed, thus lowering the fuel-bed temperature and retarding the reduction of iron to the ferrous form. Some tests have been made at the Bureau of Mines in which the gas from various points in the fuel bed was analyzed. Practically all the oxygen disappeared at a distance of 3 or 4 in. above the grate. At this point the CO content was 13 to 15 per cent and the CO_2 content 14 to 16 per cent. The atmosphere in the fuel bed at this point could scarcely be called oxidizing. It would tend to promote the formation of the more fusible ferrous silicates, a condition that would be aggravated by carrying much thicker fuel beds.

The introduction of steam under the grate is a well-known remedy for many clinker troubles.

Mr. Rigg. MR. GILBERT RIGG (*by letter*).—I wish to offer the following points in discussion of this interesting and valuable paper:

The authors state that: "In view of the fact that no softening-temperature test of the well-mixed sample of the average ash of a coal takes into account the physical distribution of the impurities in the coal, much additional investigation on this point must be made before the exact relation of these laboratory fusibility tests to clinker formation can be established."

My own work on this subject has convinced me of the truth of this statement. Both in gas producer and in boiler practice it is not at all uncommon with some coals to have the clinker form in patches, which are frequently very troublesome. A determination of the fusibility of an average sample of the ash would in such cases be liable to mislead. In making tests of this kind I prefer to burn about 5 tons of the coal on a boiler grate, and screen the resulting ash on a 2-mesh screen. The

"overs" and "throughs" are weighed separately and melting-point determinations are run on samples of the different classes of material. Under these circumstances I have sometimes found a considerable range of temperature between the melting point of the dry ash and that of the clinkered part. Mr. Rigg

This trouble is due to the irregular distribution of fluxing minerals through the coal. Pyrite streaks are familiar to everyone, and fused ferrous sulfide has a very powerful fluxing action on silicate of alumina which is present in the ashes of most coals. In many coals, if the pyrites were evenly distributed it would be insufficient to cause trouble, but occurring, as it often does, in veins and nodules, it is very liable to cause clinkering. In such cases the determination of the fusion point on an average sample of the ash would be of small value. Calcite is another flux that is liable to occur in this troublesome form.

In making fusion tests on fire clays we have a parallel set of conditions. A cone test may show a clay to be highly refractory, while bricks made from the clay may show deep pits, due to the slagging action of grains of pyrite scattered irregularly throughout the clay.

The physical nature of the clinker is also important. In trying to form an idea of the fluidity of certain slags I have been using the following method: The powdered material is formed into a $\frac{1}{2}$-in. cube, about a spiral of platinum-iridium wire. The spiral has two complete turns, $\frac{1}{8}$ in. in diameter. This is suspended in the furnace. At the softening point, the corners of the cube round off. With viscous slags the mass leaves the spiral slowly and draws out into a pear-shaped mass. Free-flowing slags run off the spiral in drops, leaving the wire nearly clean. Of course, there are all degrees of behavior between these extremes.

It seems possible that this method might furnish useful indications in connection with coal ashes.

THE NEW PHYSICAL AND CHEMICAL LABORATORY OF THE PENNSYLVANIA RAILROAD COMPANY AT ALTOONA.

By C. D. Young.

SUMMARY.

The paper treats of the activities of the Pennsylvania Railroad Company's Department of Inspection and Tests and its value in safeguarding the lives and property of its patrons. The history of the department since its establishment in 1874, and the growth which necessitated larger quarters are briefly traced.

The new laboratory building in Altoona is illustrated and described. A list of the more important features of its equipment is given, and the organization of the departments is indicated in diagrammatic form. Following a list of the materials bought under specifications, the more important general divisions of the Test Department and Chemical Laboratory work are briefly described.

The paper also contains a partial list of the publications relating to the results of tests, which have been issued from time to time by the department.

THE NEW PHYSICAL AND CHEMICAL LABORATORY OF THE PENNSYLVANIA RAILROAD COMPANY AT ALTOONA.

By C. D. Young.

Endeavoring to promote the safety of the passengers and employes on its lines by minimizing or eliminating, if possible, all accidents traceable to defective or unsuitable material, the Pennsylvania Railroad Co. has found that the quality of the material purchased for use in rails, bridges, cars and locomotives must be carefully scrutinized. Control over the quality of supplies is secured by the aid of specifications, which are based upon careful consideration of the materials available for the various uses of the railway, and by research work tending toward the development of new materials and devices, or improving those which are in general use.

Neither the reputation of the manufacturer nor a superficial inspection of the materials offered has been found to be a sufficient safeguard in the purchase of supplies, since frequently the manufacturer himself has no positive knowledge of the strength or other physical properties of the iron, steel or other metals, nor the purity of many of the articles offered for sale.

An organization with laboratories at a central point is an essential in promoting the work of thorough inspection, the importance of which is unquestioned. With this inspection, accidents to the traveling public and the employe have been reduced, and efforts in the future will be towards their further reduction. It is desirable, therefore, that the public be fully informed as to the facilities provided by one of the largest railroad companies for making tests of all its supplies and conducting investigations with a view of obtaining the best materials which can be commercially furnished.

GROWTH OF THE DEPARTMENT OF INSPECTION AND TESTS.

The Department of Tests of the Pennsylvania Railroad Co.—the first of an American railroad—has grown in the following way:

In 1874 there was established at Altoona, a department of physical tests, the organization of which was placed under the direction of Mr. Theodore N. Ely, then Superintendent of Motive Power. The first testing machine was purchased during the early part of the year. It was of 50,000-lb. capacity and was furnished by Fairbanks and Ewing. The first test was made on April 2, 1874.

In the beginning, the testing work was conducted by the Master Mechanic of the Altoona shops, but in August, 1874, the department of physical tests was placed in charge of Mr. John W. Cloud, who became the first Engineer of Tests.

A chemical laboratory, under the direction of the late Dr. Charles B. Dudley, was added in the autumn of 1875. Research work for the improvement of rails was begun, and the investigations and accumulation of experience, which later made possible the preparation of a series of "Standard Specifications," had their start.

It was not until 1879, or five years after the beginning of the testing of materials, that the physical and chemical departments were provided with a separate building. This building was a one-story frame structure, 25 by 45 ft. These quarters were soon abandoned, however, and until 1914 space was made available in a part of the shop office and storehouse building, where the departments finally occupied 15,476 sq. ft. of floor area on four floors. That the growth of the departments has been rapid is also evidenced by Fig. 1, which shows the number of employes, the number of routine physical tests, and the number of standard specifications in force for each year from 1874 to 1914. The latter quarters having become congested in the past few years, a new building with a floor area of 41,000 sq. ft., was begun in 1913 and completed in 1914. Thus, in 35 years the requirements of the departments, as shown alone by the floor space occupied, have increased more than 35 times; or, there has been an average increase of over 100 per cent for

each year since the work began. The growth of the test department and laboratory has been very much more rapid than the increase in tonnage hauled, or the extension of the general busi-

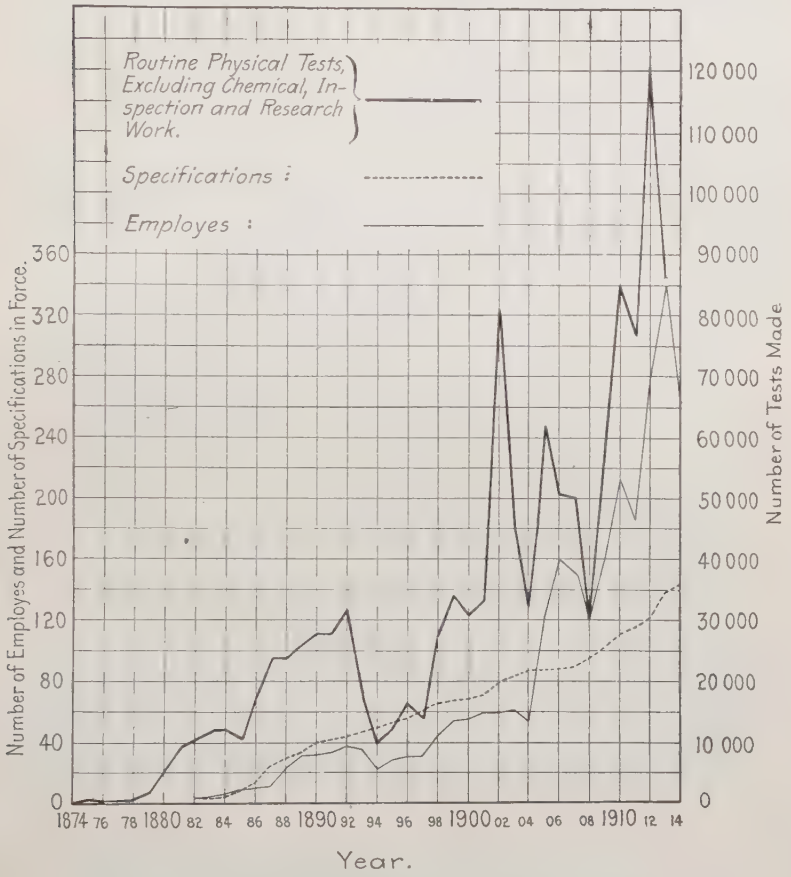


FIG. 1.—Growth of the Department of Inspection and Tests.

ness of the railroad. The reason for this is that there was almost as wide a field for the application of specifications, and the inspection and testing of materials, in the beginning as at the present time.

THE NEW BUILDING.

The new building at Altoona (Fig. 2) which has just been occupied is constructed of reinforced concrete, the reinforcement being of twisted bars. Structural steel cores are used in the concrete columns. The whole exterior is finished in red brick and red terra-cotta. It is arranged with a central service portion consisting of the middle bay which contains a stairway and an electric elevator, giving access to all parts. On the basement

FIG. 2.—The New Building.

floor of the service section there is a receiving room for materials (Fig. 3). This room communicates with the elevator for the distribution of small samples to the different sections of the building, while large pieces may be lifted to the physical-test section by means of a 10-ton traveling crane with a hatchway in the main floor, as shown in Fig. 4. There is a machine room in the basement and in this all of the metal test specimens are prepared. On this floor there are two large fireproof vaults for the storage of letter files and the like, and a room for chemical stores.

Description of Each Floor.—The first or street floor (Fig. 4) is devoted to physical tests. It contains a physical laboratory with five universal tension and compression testing machines, the largest of which has a capacity of 1,000,000 lb., and all are served by the traveling crane. On this floor are separate sections for oil, cement and lagging, hose, rail, miscellaneous and heat-treatment tests.

The second floor (Fig. 5) is used for office, locker and toilet rooms, the south end being occupied by the office force of the Engineer of Tests and the north end by that of the Chief Chemist.

The third floor (Fig. 6) is divided into laboratory rooms for bacteriology, rubber, water and gas analyses, photometry and lamp tests, and the calibration of electric instruments.

The whole fourth floor (Fig. 7) is used as a general chemical laboratory with a separate chemical balance room. The central bay is extended up to form a fifth floor (Fig. 8) and comprises a photographic studio and dark room, while the roof of the remaining portion of the building is used for experimental work and tests where exposure to the atmosphere is required.

Lighting.—Direct lighting with tungsten lamps is the system of illumination. "Abolite" metal reflectors are used in the basement and on the first floor, with "Pyro" glass reflectors on the second or office floor. In the chemical laboratory, where metal would be injuriously acted upon by gases, "Holophane" glass reflectors are in use. All of the lighting and power conduits were placed in the floors before pouring the concrete. Telephone, dictaphone and buzzer systems are installed in the floor conduits, and in addition great flexibility is possible in the location of these fixtures by the use of a chair rail around the walls of each room, the chair rail having three separate grooves for wires.

Heating.—The building is heated by direct steam radiators with a single pipe system, and the radiators are placed under the windows. A hot-water service, with a heating and circulating tank in the basement is provided. The gas, steam, air, water and hydraulic lines are of open work, and all pipe risers are in a common conduit in the central service bay of the building.

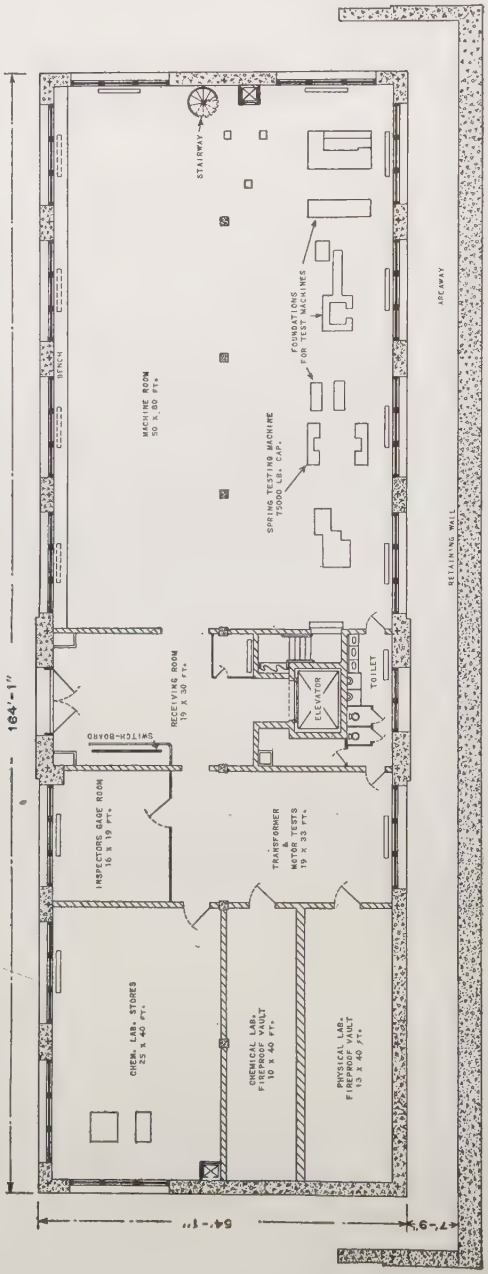


FIG. 3.—Basement Plan.

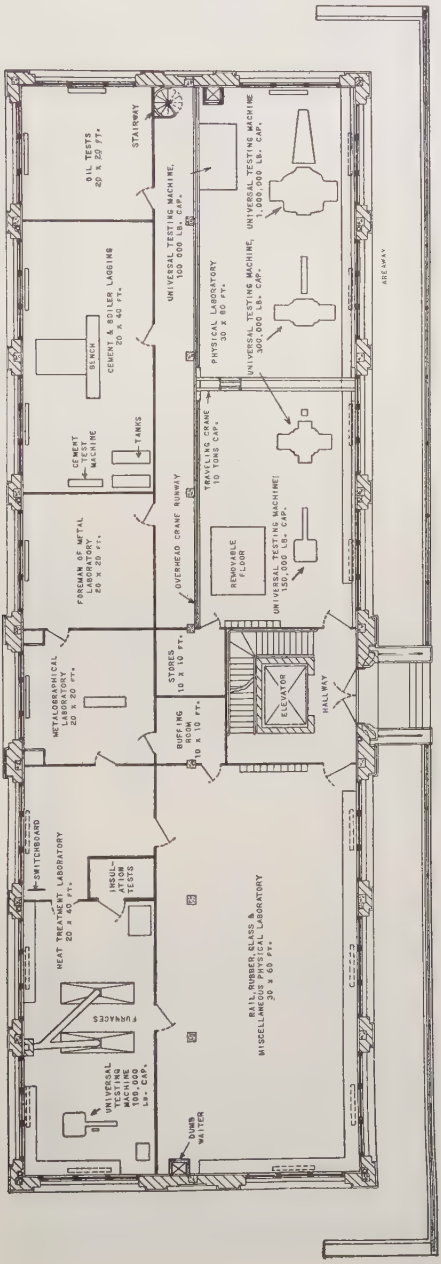

OIL TESTS 20 X 23 FT.
STAIRWAY
UNIVERSAL TESTING MACHINE 100 000 LB. CAP.
PHYSICAL LABORATORY 30 X 80 FT.
UNIVERSAL TESTING MACHINE 300,000 LB. CAP.
UNIVERSAL TESTING MACHINE 1,000,000 LB. CAP.
AREAWAY
BENCH
CEMENT & BOILER LAGGING 20 X 40 FT.
CEMENT TEST MACHINE
TANKS
TRAVELING CRANE 10 TONS CAP.
FOREMAN OF METAL TESTING DEPT. 20 X 20 FT.
OVERHEAD CRANE RUNWAY
REMOVABLE FLOOR
UNIVERSAL TESTING MACHINE 150,000 LB. CAP.
STORES 10 X 10 FT.
BUFFING ROOM 10 X 10 FT.
METALLOGRAPHICAL LABORATORY 20 X 20 FT.
ELEVATOR
HALLWAY
SWITCHBOARD
HEAT TREATMENT LABORATORY 20 X 40 FT.
INSULATION TESTS
FURNACES
UNIVERSAL TESTING MACHINE 100,000 LB. CAP.
DUMB WAITER
RAIL, RUBBER, GLASS & MISCELLANEOUS PHYSICAL LABORATORY 30 X 60 FT.

FIG. 4.—First-Floor Plan.

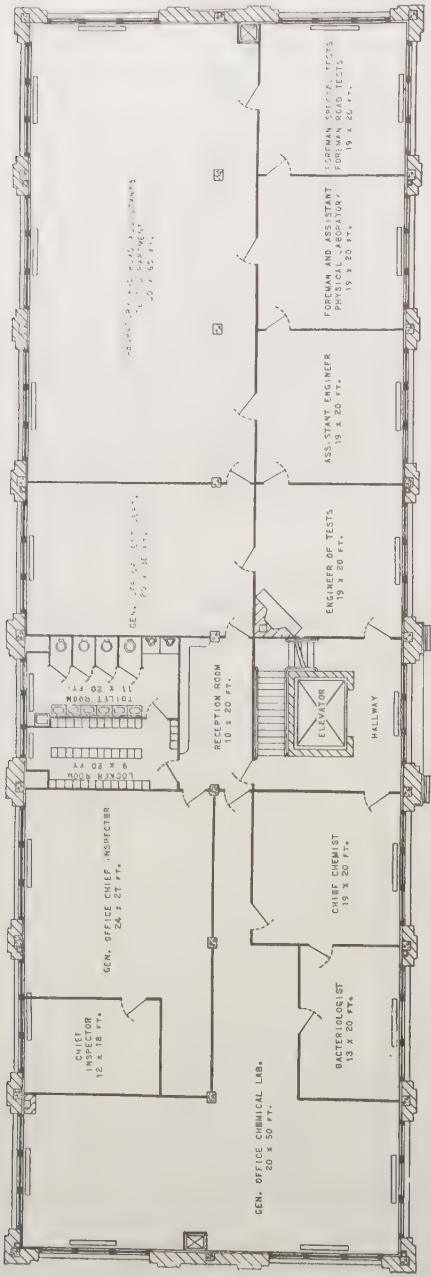


FIG. 5.—Second-Floor Plan.

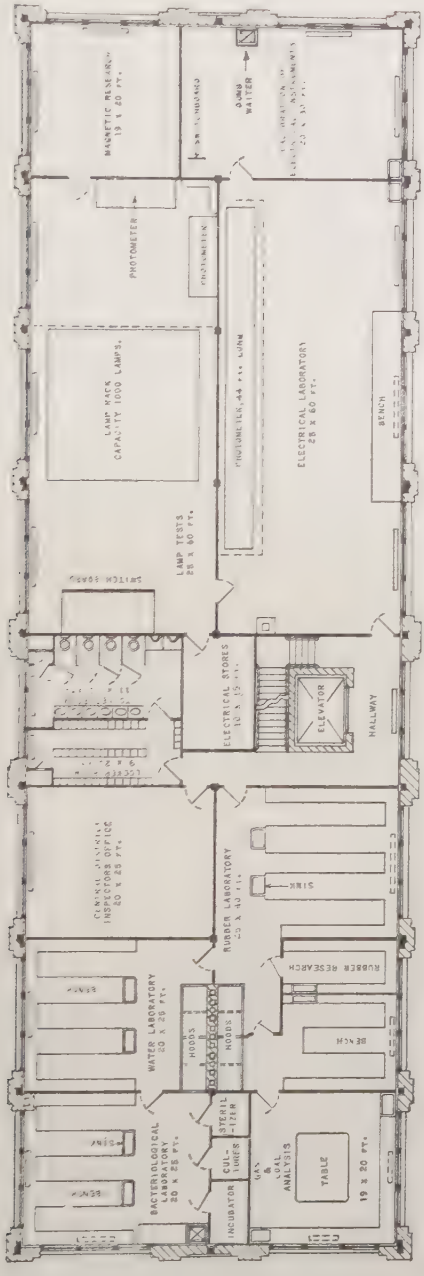


FIG. 6.—Third-Floor Plan.

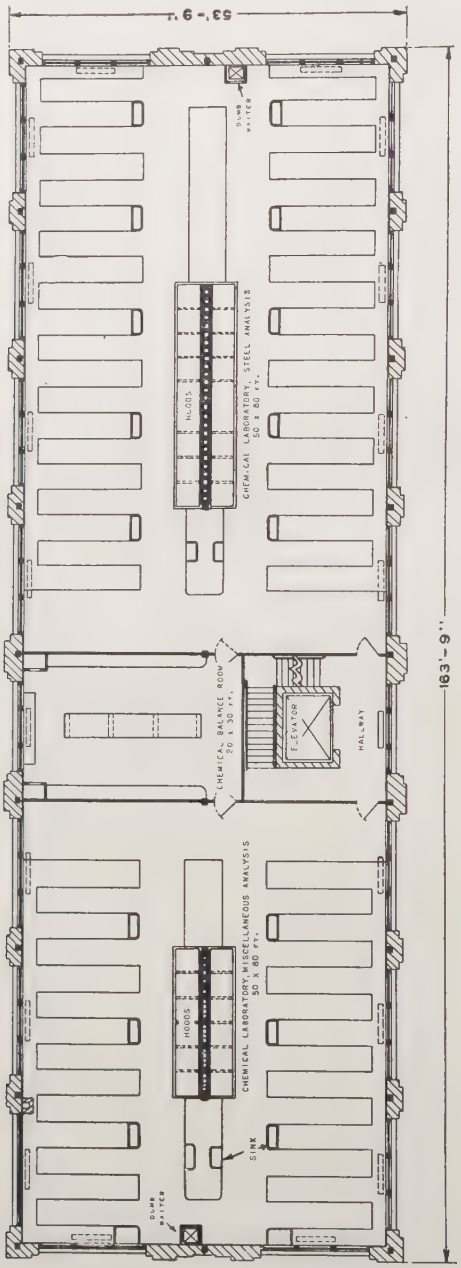


FIG. 7.—Fourth-Floor Plan.

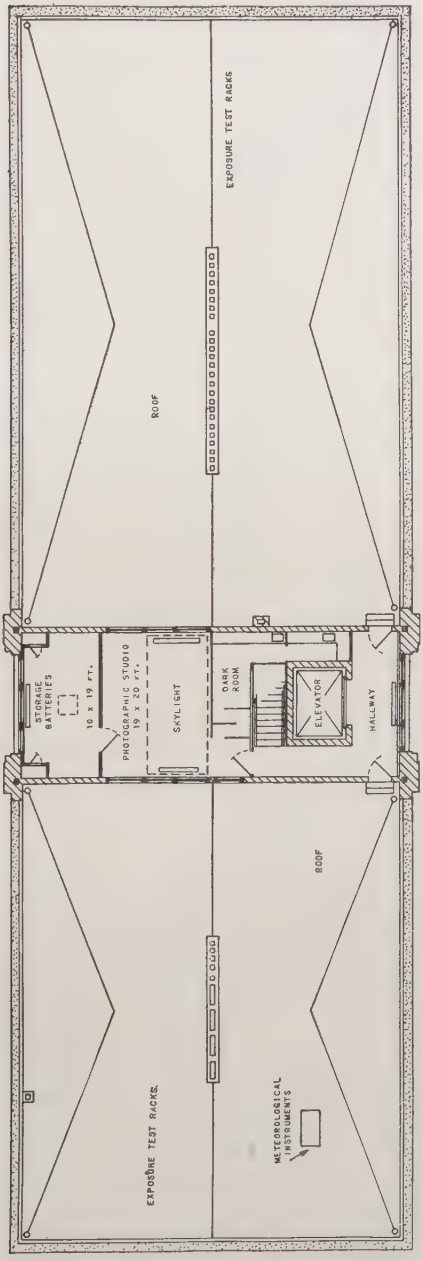


FIG. 8.—Fifth-Floor Plan.

Interior Finish.—The interior of the building is finished in natural chestnut throughout, with the exception of the office rooms, which are finished in imitation mahogany. All interior doors and partitions are glazed.

Floors.—The floors, with the exception of the basement where the floor is of concrete and the physical laboratory where it is of wood on concrete, are of magnesium-cement composition.

Cost.—It is noteworthy that the building was constructed and equipped complete within the original estimates and appropriation. The building itself cost about $150,000. An estimate of the value of the contents is as follows:

Physical laboratory	$100,000
Chemical "	25,000

With equipment complete, the investment for the laboratories is about $275,000.

PHYSICAL LABORATORY.

Among the machines and apparatus that compose the equipment of the physical laboratory, there are the following:

Six universal tension and compression testing machines, one of 1,000,000, two of 300,000, two of 100,000-lb. and one of 150-000 lb. capacity;
One vibratory endurance spring testing machine of 75,000-lb. capacity;
One 43-ft. drop-testing machine;
Two vibrating stay-bolt, testing machines;
One Brinell hardness testing machine;
One 2000-lb. cement testing machine;
One horizontal microscope, with camera for metalographic work;
One grinding, buffing and etching outfit for the preparation of samples for microscopic work.

In the machine room, where the sample test specimens are prepared, the following tools are used:

Two 14-in. engine lathes;
One 12-in. drilling lathe;
One 24-in. shaper;
One 24-in. radial drill;
Two milling machines for specimens;
One 30-in. cold saw;
Two motor hack saws;
Two tool grinders.

For the work in testing air-brake, signal and tank hose and other miscellaneous tests including steam and hydraulic gages, there are:

Six rubber stretching machines;
One friction test rack for rubber;
One hose mounting machine;
One vibrating test rack for hose;
One continuous test rack for rubber;
Four tension testing machines for rubber;
One stretching machine for rubber insulation;
One spring micrometer machine;
One vacuum gage testing machine;
One arbor press specimen cutter;
One hydraulic gage testing machine, capacity 25,000 lb. per sq. in.
One dead-weight gage testing machine, capacity six gages;
One wiggling testing machine for hose;
One bumping testing machine for gages;
One whipping testing machine for gages;
One hydraulic machine for testing gage glasses.

The materials for test, including samples which have been obtained by the inspectors at outlying points and those sent to the department by the shops, are brought into the building through the receiving room (Fig. 3). They are distributed throughout the building from that point, the metal specimens going to the machine room in the basement for preparation, then to the physical laboratory for tension, compression, vibratory or other tests, and to the chemical laboratory for analysis.

Rubber, Air-Brake Hose and Miscellaneous Laboratory.—The extent of the work of this department is indicated by the fact that the needs of the railroad are about 635,000 pieces of air-brake hose per year. There are now being installed machines for air-brake, signal and tank hose, and other miscellaneous tests, including steam and hydraulic gages, and gage glasses for boilers and lubricators.

Heat-Treatment Laboratory.—This department, on the first floor (Fig. 4), is for the development of standards in the heat treatment of metals during the process of their manufacture for use in railway equipment. Investigations are carried out to study the effect of various heat treatments on a large variety of carbon and alloy steels. They are also made to determine

the properties of non-ferrous alloys, including the coefficient of expansion. Shop-manufactured locomotive and car springs, involving as they do a form of heat treatment, are sampled and tested regularly to determine their acceptability for service.

Large castings of various kinds have been heat-treated by this department with the aid of outside facilities with a gratifying degree of success. The effect of chemistry and heat treatment upon the endurance of materials to repeated stresses is tested out by revolution and vibration tests, including vibration tests on complete springs. Rails, splice bars and tie plates are heat-treated to study the increased service it is possible to secure. The effects of heat treatment are noted and a wide range of working conditions are applied on a variety of high-speed tool steels to ascertain the best chemical characteristics.

Investigations are made on various types of fireproof material for the purpose of maintaining a high standard. The testing of felt and insulating papers used for lining refrigerator cars has been made necessary by the large variety of materials of this kind on the market, the keen competition among manufacturers, and the ease with which the highest grade and best materials can be closely imitated by cheap and inferior products. This laboratory is equipped with an insulated room and electrical heating arrangements for this work, the tests being designed to represent as nearly as possible the service conditions to which these materials would be subjected. Temperature measurements are made of various types of refrigerator-car construction by means of resistance thermometers. Aside from the measurements of high temperatures in the laboratory, periodic calibrations are made of the various pyrometers. The heat-treatment department in general carries on a large variety of special work, and there is but little that falls without its range of possibilities, even to the extent of heat-treating glass ware.

ELECTRICAL LABORATORY.

Lamp Tests.—On the third floor (Fig. 6) the equipment for lamp tests consists of three photometers, a lamp test rack of 1000 lamps capacity, with switchboard, transformers and potential regulator equipment. This work was taken up in 1902 with a view of obtaining data for the preparation of specifications to

secure uniformity in the ordering of incandescent lamps, and the maintaining of sufficiently high standards. It consists mainly of life tests of lamps at abnormal voltages and tests for the efficiency of illumination, as well as the investigation of new developments in the general field of illumination as applied to railway work.

Standardization of Instruments.—A division of the electrical laboratory is employed in investigations and development work along electrical lines, and the standardization of electrical instruments. Part of this work is done at the laboratory, and part of it, when necessary, at other points, by laboratory men. The character of the work may be judged from the following examples upon which comprehensive reports have been made:

An investigation of electrolysis in systems of underground metallic structures;
Tests and investigations of the construction of various makes of transformers;
Tests of various makes of primary and secondary battery cells;
Oscillographic tests for linear and angular velocity, wave forms, etc.;
Investigations of special cases of electrical troubles;
The development of an electrical method of measuring the hardness and homogeneity of steel.

Matters such as these are reported on and recommendations made. Electrical instruments are sent in from all points on the Pennsylvania System to this department for calibration and repair, and men from the laboratory are sent out to inspect and check electrical instruments on switchboards at the various power plants, and at other points.

LABORATORY AND ROAD ASSISTANTS.

The large room on the second floor (Fig. 5) is provided for the force of laboratory and road assistants coming under the direction of the foremen of Road Tests and Special Tests. The duties of these men are varied, and include tests of locomotives on the road or tests of equipment with special devices; the tonnage rating of trains and the following up of all experimental appliances which are put into service for test purposes.

Photographic Studio.—The fifth floor (Fig. 8) has been arranged for photographic work, consisting largely in making prints of metal sections, photomicrographs of steel rails forming

a large part of these. Photographs of parts which have failed in service are also made for convenient preservation and study. The photographic work requires the services of two men and about 25,000 prints per year are made.

CHEMICAL LABORATORY.

Metallurgical Work.—The main chemical laboratory on the fourth floor (Fig. 7) is divided by the central balance room into two departments, the larger one of these being devoted exclusively to metallurgical chemistry. In this department methods are developed for the determination of the elements in plain-carbon steels, alloy steels, and non-ferrous alloys used for bearing backs and linings, packing-ring metal for different purposes, etc. Data are obtained leading to the development of specifications for this class of products, and samples of shipments are analyzed to determine whether they are acceptable under the specifications adopted. This steel laboratory has facilities for analyzing 100,000 samples per year.

Miscellaneous Work.—The smaller of these two laboratories is for work of a more general character, being used for the examination of fuels, the development of specifications for paint products, lubricating and burning oils, boiler compounds, lacquers, plush, car cleaners, cutting compounds, belt dressing, polishing compounds, hydraulic-jack liquids, fuses, track caps, fire-extinguishing preparations, the recovery of used or wasted products, etc.

In both of these laboratories much time has been spent in the examination of broken or "failed" parts of equipment, in an effort to determine the cause and with a view to the prevention of accidents which, aside from the money losses, might result in injuries or loss of life.

Certain food products, used in the dining-car service, are examined at times; and other miscellaneous investigations are made, as of conditions which may have led to loss from the damage of freight in transit, and to so establish methods for preventing such loss. During the past year a considerable amount of work has been devoted to the chemistry of tunnel air in connection with the installation of ventilating systems.

The total list of activities touched upon would be too long for enumeration in a paper of this character.

The chemical analysis of rubber compounds has been studied and much experimental work done in perfecting a method whereby material of this kind can be bought on specifications which define and limit its chemical properties. At present there is in force a specification for high-grade rubber insulation.

Samples from all shipments are analyzed and at the same time experimental work is being carried on to improve the method of analysis, and to devise others so that specifications may be drawn covering the chemical properties of all grades of rubber materials.

Manufacturing Laboratory.—A manufacturing laboratory, which might be called a small factory, is maintained in a separate building which is under the direct supervision of the Chief Chemist, and new products are manufactured in this until such time as it is found advisable to purchase them from "outside" manufacturers.

Laboratory Car.—In addition to the steel-rail work at Altoona a laboratory car has been built to be moved, as required, to that point where steel rails in process of manufacture are to be inspected. The object in equipping this car (Fig. 9) is to make chemical analyses of the finished rails at the mills by a force of chemists under the Chief Chemist. This, it is expected, will avoid delays which at times occur in the operation of the mills, and are impossible to avoid without the facility of a suitable force at hand when the rolling is taking place, in order to keep up with the chemical requirements of the company's specifications. The car is equipped with furnaces for combustion and all other necessary apparatus for general chemical work in connection with the inspection of steel rails.

Bacteriological Laboratory.—When the department of chemistry was established, problems were frequently presented which applied chemistry could not solve satisfactorily. It was found, for example, that a chemical examination of water might show the presence of organic constituents, but it was impossible to tell the source of these. A water might contain a large amount of organic material of vegetable origin and yet not carry any infectious material which would likely give rise to disease, while other

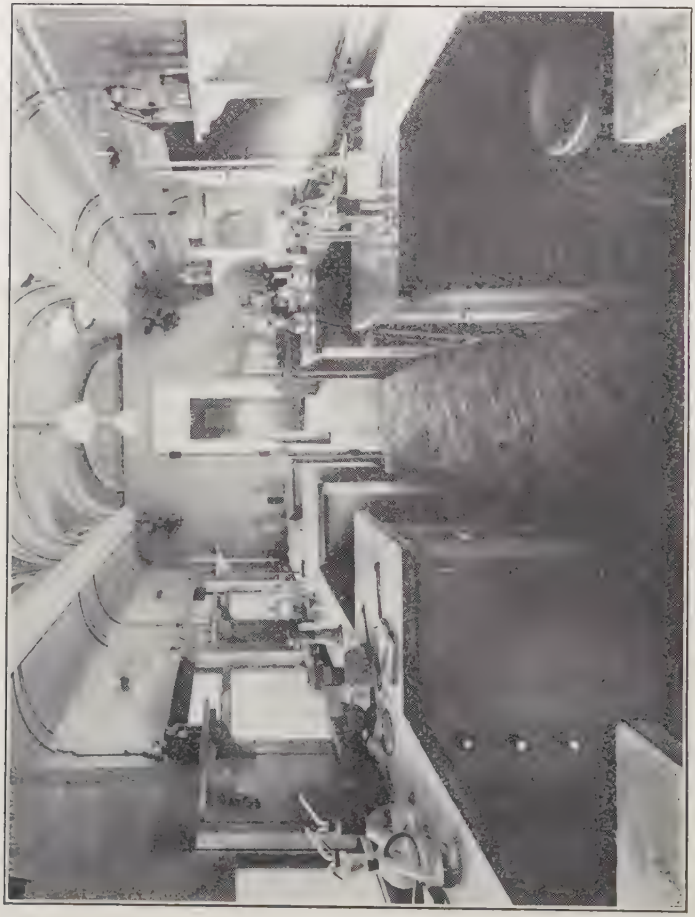


FIG. 9.—Interior of Laboratory Car.

samples low in organic constituents were believed to carry infectious germs which might render their use very dangerous to employes or patrons of the road.

It was also found necessary to supervise certain sanitary matters and to disinfect cars, offices and waiting rooms under certain conditions, but it was not known what disinfectants were destructive to specific disease-producing bacteria. Manufacturing concerns were offering various disinfecting preparations, but the officers of the company had no means of determining which ones were efficient and the problem could not be solved by chemistry alone. These questions were considered so important that it was decided that a division of bacteriological chemistry was necessary, and on November 1, 1899, such a laboratory was established.

The work in bacteriology and water analysis has increased constantly, and at the present time four men are employed in the laboratory. The department cooperates with the Surgeon General of the United States in the enforcement of the quarantine regulations of 1913, which require that railroad companies shall furnish wholesome drinking water and proper ice supply to passengers using their cars. It might be a satisfaction for the public to know that water which contains anything indicative of injurious contamination is not permitted to be introduced into the drinking containers of the Pennsylvania coach.

The department regulates the standardization of disinfectants and issues instructions concerning their application for the protection of passengers and employes, as well as the disinfection of stock cars. Special care is taken to prevent any infected employe from coming in contact with the public.

In 1914 bacteriological and chemical examination were made of 609 samples of drinking water. There were 3112 bacteriological examinations of pathological specimens, submitted by the relief association physicians. The total number of bacteriological examinations was 3621, or an average of more than ten per day.

In addition, this department has under its care the examination of boiler feed waters and the formulation of methods for their treatment. In 1913, examinations of 287 boiler feed waters were made, while in 1914 the number was 282.

DYNAMOMETER CAR.

As part of the equipment of the Test Department there is a dynamometer car (Fig. 10). This car which was built in 1906, is the fifth of a series of such cars which have been in use on the Pennsylvania railroad.

LOCOMOTIVE TESTING PLANT.

There is a locomotive testing plant (Fig. 11) which is located adjacent to the test department building. This plant was erected in 1905, after having been in use at the St. Louis Exposition in 1904, and is operated by a force of 26 men.

BRAKE-SHOE TESTING MACHINE.

There is being installed in a separate building a brake-shoe testing machine which will be the first of its kind, in that it will have two dynamometers of 4000-lb. capacity, which will make it possible to obtain the coefficient of friction of brake shoes when two shoes are applied to a single wheel (clasp-brake conditions). The car wheel will run upon an idler wheel, representing the action of a rail upon the wheel.

THE EXTENT AND VARIETY OF THE MATERIALS TESTED.

The scope of the work now embraced by these departments coming under the jurisdiction of Mr. J. T. Wallis, General Superintendent of Motive Power, at Altoona, can be better appreciated when it is understood that the cost of the materials covered by the inspection and tests, and entering into the construction of the railroad rolling stock and track, in 1913 amounted to $82,119,480, while the cost of operating the test department and chemical laboratory for the same year was $534,060. This figure for the cost of operation covers all supervision, supplies and the less important new apparatus added at times and not chargeable to the usual railway A. & B. account. The cost of operating the dynamometer car and the locomotive testing plant is also included. Interest on investment, depreciation of plant and insurance are not included.

For an approximation and using these figures, it is interesting to observe that the total cost of operating the departments, including all additional work and inspection, is about 0.6 per cent of the cost.

FIG. 10.—Interior of Dynamometer Car.

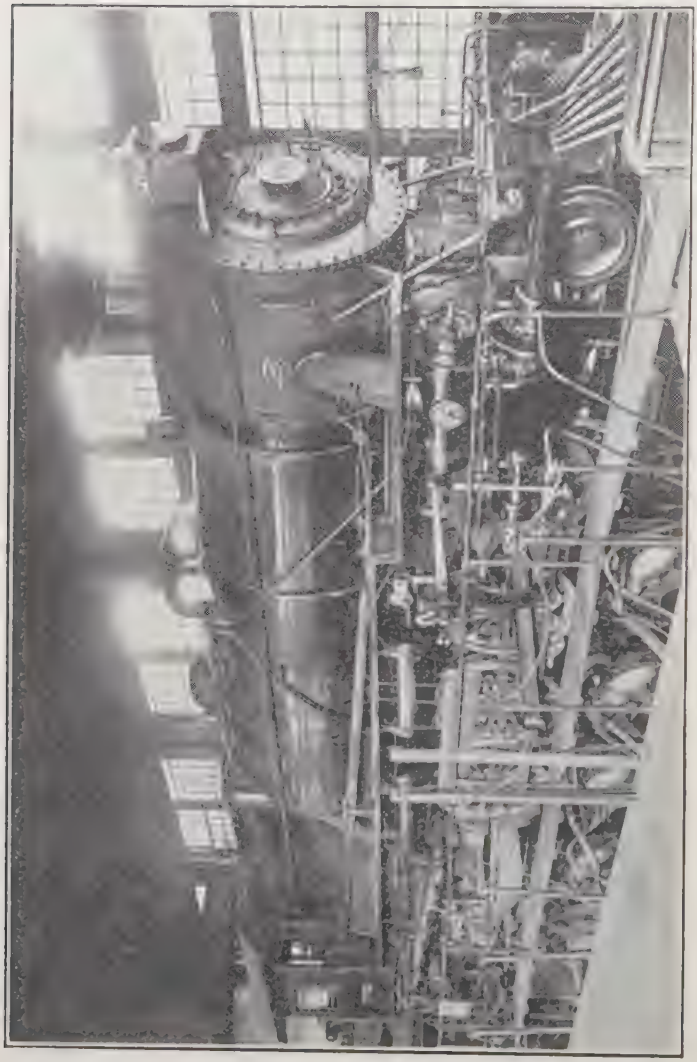


FIG. 11.—Locomotive Testing Plant.

The year 1913 was perhaps a record one for the test department and laboratory, and the extent and variety of the work of the departments can be shown by a few examples for that year. There were 61,148 separate reports of material tests issued by the test department. In the physical laboratory, while no record was kept of the number of samples examined, 138,886 tests were made. These tests represented quantities such as the following:

Of bar iron 149,863,693 lb. were tested and 6,246,611 lb. rejected; of staybolt iron, 15,385 tests representing 8,301,960 lb. were made; of cement, 29,231 tests were made, representing 587,900 bbl., of which 13,600 bbl. were rejected; of wheels, 310,381 were inspected, and 1213 were rejected; of axles 164,810 were tested and 8035 were rejected; 290 samples, representing 56,322 yds. of plush, were tested; of air-brake hose, samples representing 634,807 were tested and 84,826 rejected.

In the chemical laboratory, during 1913, a total of 57,309 samples were analyzed, involving about 286,545 determinations.

The materials which are now brought under specifications and which must be passed upon by the test department or the chemical laboratory are as follows:

Asphaltum.
Axles, 4 kinds.

Barrels, metal.
Bearings.
Belting.
Blue Vitriol.
Bolsters, cast-steel.
Brake beams.
Brake shoes.
Brooms.

Car roofing.
Cars.
Castings, steel.
Caustic soda.
Chain.
Conduit, steel.
Copper, ingot.
Copper, phosphor.
Couplers.
Crank pins.

Disinfectant.

Electrolyte.

Frames, locomotive.
Fuses, 2 kinds.

Hose, air-brake.
Hose, miscellaneous.
Hose, signal.
Hose, steam.
Hose, tank.
Hose gaskets.

Iron, merchant bar.
Iron, stay-bolt.

Jack shafts.

Knuckles.

Lagging, magnesia.
Lamps, incandescent.
Lard oil, 2 kinds.
Lead, pig.
Lubricator glasses.
Lumber.

Matting, rubber.

Packing, journal-box.
Paint, cabin-car.
Paint, freight-car.
Paint, Indian red.
Paint, passenger-car truck.
Paint, refrigerator car.
Paint, standard black.
Paint, standard building.
Paint, telegraph cross-arm.
Paint, tuscan red.
Paper, refrigerator car.
Petroleum products, 5 kinds.
Pipe, steel.

Pipe, wrought iron.
Pivot pins.
Plush, 3 kinds.
Sal Ammoniac.
Soap, 3 kinds.
Soda ash.
Sponges.
Springs, elliptical.
Springs, helical.
Steam gages.
Steam-pipe covering.
Steel, 21 kinds.
Steel, plate-coated, 3 kinds.
Sulfuric acid.
Tallow.
Thermometers.
Tin.
Tires, driving.
Torpedoes.
Truck sides.
Tubes, boiler.
Turpentine.
Waste
Water gage glasses.
Wheels, cast-iron.
Wheels, rolled-steel.
Wire and cable.
Wood creosote.
Wood preservative.
Wool, mineral.
Zinc.

During 1913 there were inspected, while building at manufacturers' works, 24,966 freight cars, 343 steel passenger cars, and 190 locomotives. The value of the materials rejected through the test department in 1913 was as follows:

Physical Laboratory	$776,928
Chemical Laboratory	65,767

ORGANIZATION OF DEPARTMENTS.

As outlined in the diagram of the organization (Fig. 12), the inspection at the manufacturer's works and the collection and forwarding of samples to Altoona is carried out under the direction of the Chief Inspector, with permanent resident inspectors and forces for the central district at Altoona, the western district at Pittsburgh and the eastern district at Philadelphia. In addition, when equipment is being built at outlying points, temporary inspection forces are maintained at these places during the progress of the work.

As previously stated, the work of the department began under the direction of Mr. John W. Cloud. In May, 1879, he was appointed the first Engineer of Tests and continued under that title until July, 1886, when he succeeded to the office of Mechanical Engineer, retaining control of the test department. Mr. Axel S. Vogt, the present Mechanical Engineer, succeeded Mr. Cloud in March, 1887. The work of the department under the Mechanical Engineer was in direct charge of Mr. W. O. Dunbar from July, 1886, to July, 1893. From the latter date to July, 1903, the Assistant Mechanical Engineer had direct charge of all the work of the department. During this latter

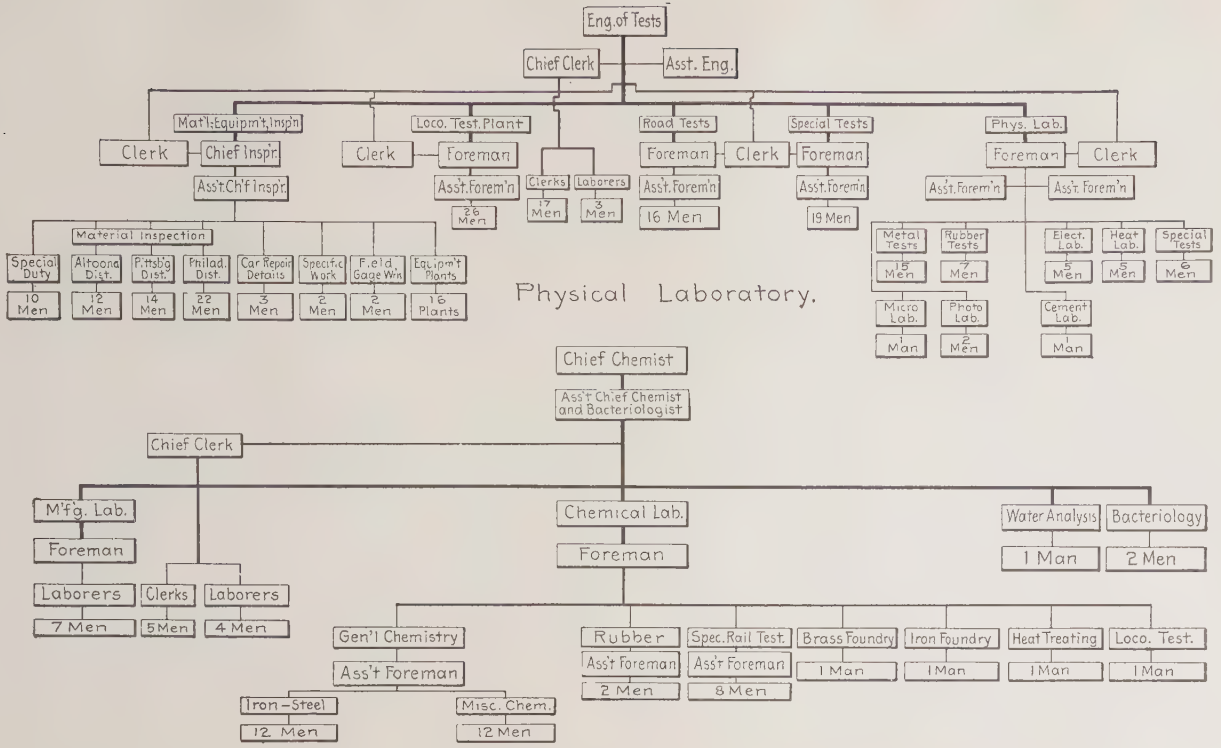


FIG. 12.—Organization of Department.

period the Assistant Mechanical Engineers were, Mr. A. W. Gibbs, from July, 1893, to August, 1902, and Mr. W. F. Kiesel, from the latter date until July, 1903. In August, 1903, Mr. E. D. Nelson was appointed Engineer of Tests, and in September, 1911, was succeeded by the writer.

Two men have been in charge of the chemical laboratory, Dr. Charles B. Dudley from November, 1875, until his death, December 10, 1909. Since December, 1909, Dr. F. N. Pease has held the position.

PUBLICATIONS.

That the information collected and the developments which have been made in the chemical laboratory and the test department have been freely given to the public, is well exemplified by papers and addresses which have been presented by the late Doctor Dudley.[1]

In addition to the works of Doctor Dudley, there have been published by the test department, 27 printed bulletins covering field tests and the work of the locomotive testing plant. These bulletins contain 3260 pages of diagrams, tabulations and printed matter, and, with the exception of those which directly treat of the commercial properties of some concerns, have been distributed to the public without cost. Some of the more noteworthy of the latter bulletins are the following:

Bulletin No. 5, Tests of a Class E2*a* Locomotive;
Bulletin No. 10, Tests of a Class H8*sb* Locomotive;
Bulletin No. 11, Tests of a Class E3*sd* Passenger Locomotive;
Bulletin No. 13, The Smokebox Superheater;
Bulletin No. 16, Fuel Economy Tests;
Bulletin No. 18, Tests of a Class K2*sa* Passenger Locomotive;
Bulletin No. 21, Tests of a Class E6*s* Passenger Locomotive;
Bulletin No. 22, Comparison of Passenger Locomotives;
Bulletin No. 23, Piston-Valve Diameter and Valve-Stem Stresses;
Bulletin No. 24, Superheater Tests;
Bulletin No. 25, Brake Tests;
Bulletin No. 26, Train Resistance and Tonnage Rating;
Bulletin No. 27, Tests of a Class E6*s* Locomotive.

[1] "The Life and Life Work of Charles B. Dudley," Am. Soc. Test. Mats., 1910.

Further contributions by the department are "The Painting of Steel Passenger Cars,"[1] "Locomotive Superheaters and their Performance."[2]

With the facilities described and the excellent organization built upon the experience of over forty years, it is apparent that the Pennsylvania Railroad Co. is in position to exercise the closest supervision over its purchases and supplies. At the same time it is capable of not only keeping abreast with the developments of materials of construction, methods of health preservation and safety of railway operation, but should, as it has attempted to do in the past, be in the lead in dealing with the many and diversified technical problems continually arising.

[1] The author, *Journal*, Am. Soc. Mech. Engrs., April, 1913.
[2] The author, *Journal*, Franklin Institute, July and August, 1914.

A UNIVERSAL STRAINOMETER OF SIMPLE DESIGN.

By S. H. Graf.

SUMMARY.

The following paper presents to the Society a form of extension and compression instrument possessing the following advantages:

1. Applicability to a wide range of specimen sizes, either for tension or compression;
2. Ease of use with a satisfactory degree of accuracy;
3. Simple construction and low cost.

The device combines in one instrument all the requirements usually met less satisfactorily by several extensometers and compressometers, and it is believed that it will be especially valuable to commercial and municipal laboratories of limited means, where the outlay of capital for the several instruments usually required is not justified.

A UNIVERSAL STRAINOMETER OF SIMPLE DESIGN.

By S. H. Graf.

In the municipal, commercial, or college laboratory for testing materials a wide range of work is usually handled, and specimens of sizes other than the ordinary standard ones must often be tested. Although most of the ordinary routine tests are made without deformation measurements within the elastic limit, these data are sometimes required, making it necessary in the past to have at hand at least the following:

1. An extensometer for 2 and 8-in. gage lengths;

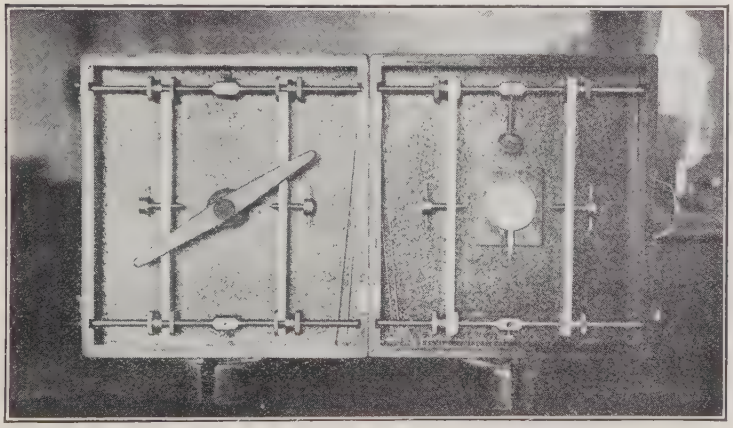


Fig. 1.—Universal Strainometer in Case.

2. A compressometer for small specimens, with special collars;

3. A compressometer for 6 or 8-in. concrete cylinders.

It is believed that a simple, direct-indicating, universal instrument for the measurement of both extension and compression would serve all ordinary purposes of the commercial and city laboratories.

The writer and his associates, Messrs. C. L. Knopf and R. B. Boals, have experimented for several years with various

arrangements to accomplish the purpose indicated and have finally arrived at the device shown in Fig. 1.

Briefly stated, the instrument consists of two simple adjustable frames, each carrying two screws bearing on the gage marks on the specimen. One of the frames carries an Ames dial and pivots on a rod held rigidly in the other frame, the dial indicating twice the actual deformation. A toggle clamp prevents the frame from separating from the pivot, and a slender steel rod actuates the staff of the gage head. The error due to tilting of the dial frame within the range of any test is of no consequence, and within the elastic limit it is not a readable quantity.

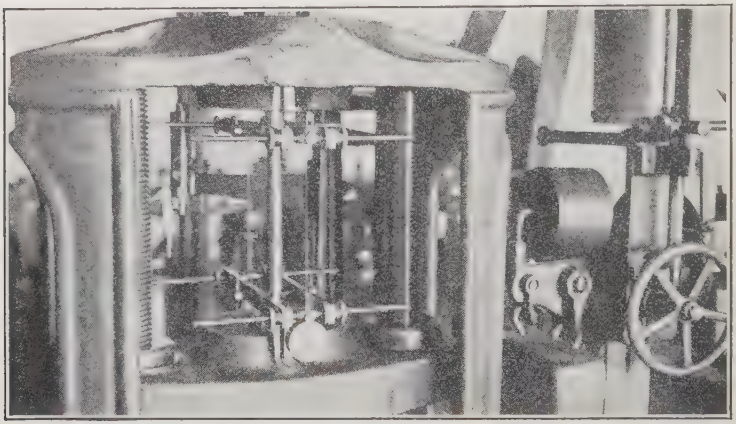


FIG. 2.—Strainometer Used as Extensometer.

Figs. 2, 3, and 4 show the instrument applied to various specimens and indicate its range; this range includes specimens either in tension or compression up to 8 in. in diameter or square, and of any gage length from 2 in. up. To adapt the instrument to different gage lengths it is only necessary to use pivot and dial rods of different lengths; for the ordinary lengths of specimens, rods of drill steel $\frac{1}{8}$ and $\frac{1}{16}$ in. in diameter, respectively, are suitable, while for special tests where the length may be considerable, light wooden strips with steel inserts in the ends are perhaps most satisfactory.

In addition to its universal applicability this strainometer also possesses the following advantages:

1. Ease of use; no special skill being required to read the dial. Dial is zero setting so that there is no troublesome zero reading to subtract; the reading divided by two give the net deformation.

2. Readings may be taken "on the run" with accuracy, even by an inexperienced observer. Fig. 5 shows load-deformation diagrams plotted from readings actually taken "on the run" by students who had never before read an extensometer or compressometer of any kind.

3. A change in rate of deformation from any cause is at once plainly apparent.

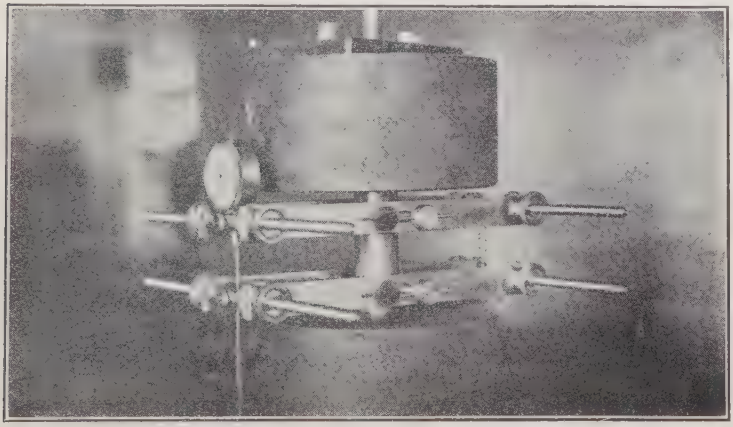


FIG. 3.—Strainometer Used in Compression Test of a Small Cast-Iron Specimen.

4. The instrument may be easily and quickly readjusted and applied to any specimen within its range. It is simple, and with ordinary care does not get out of order.

5. It may be constructed by any good mechanic. The only delicate part is the indicating mechanism, consisting of the ordinary form of 0.3 by 0.001-in. Ames dial with plain back and celluloid crystal, which may be purchased complete and ready to attach.

6. The cost of the instrument complete is low.

The accuracy and reliability of the Ames dial when applied to strain measurements has been well established, and it is not

necessary here to cite calibration data for the dial. Repeated calibrations of the complete strainometer as just described, both against a micrometer and against test bars of known modulus, have shown the instrument to be fully as accurate as others designed to read to 0.0001 in. Some of the extensometers and compressometers on the market, while fundamentally of very precise design, are so complicated and cumbersome, as well as slow and difficult to read, that their apparent accuracy as shown by calibration cannot be obtained under operating conditions.

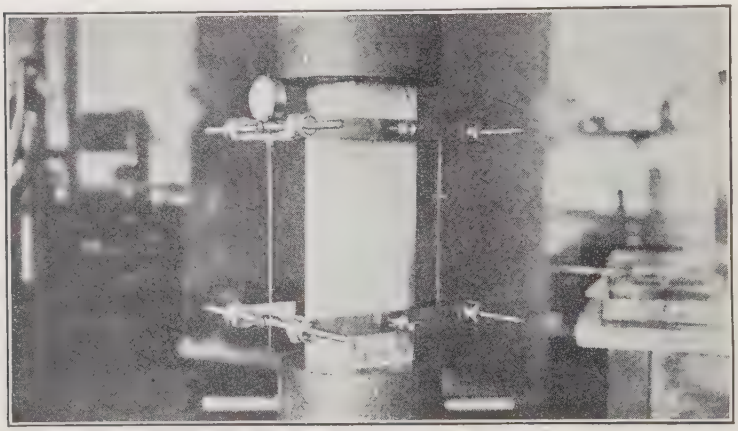


FIG. 4.—Strainometer as Arranged for Compression Test of Wood or Concrete.

Some objection has been made to instruments having only two (instead of three) points of attachment, but numerous studies made by means of the Berry strain gage on the distribution of stress in various specimens under test have convinced the author that under proper conditions of gripping tension specimens and of bedding compression specimens, the two-point instrument will, with equal care in centering, give the average deformation as faithfully as the other. If provided with three dials or micrometers the three-point instrument is useful in showing roughly the distribution of stress, but this makes the instrument too complicated for all ordinary purposes. Three observers would be required if readings were to be taken

on the run, and even when load is applied by increments, it is difficult for a single observer to read three dials or micrometers accurately.

The strainometer as described has now been in use for nearly a year in the laboratory at the Oregon State Agricultural College, and has proved very satisfactory, not only for class work, but also for commercial and investigational work.

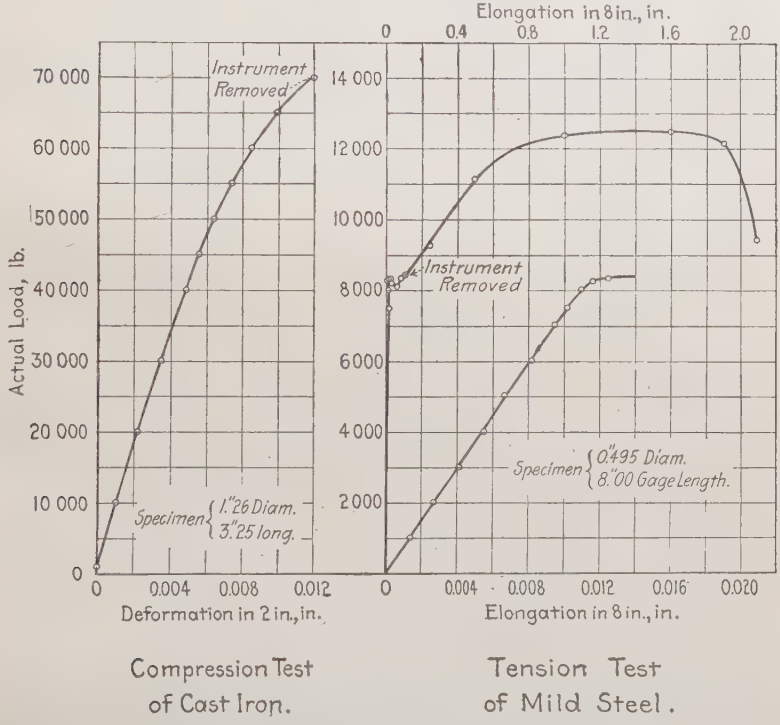


FIG. 5.—Load-Deformation Diagrams. Readings Taken "on the Run" by Inexperienced Operators.

In conclusion, the author wishes to say that he and his associates claim no great originality for this strainometer, for it really embodies a combination of principles previously applied in various other instruments. Since it has satisfactorily filled a need in the author's laboratory, however, it is offered to the Society in the hope that the idea will be of value to others.

A LABORATORY METHOD OF DETERMINING PRESSURES ON WALLS AND BINS.

By J. Hammond Smith.

SUMMARY.

The paper describes an apparatus for determining the point of application, line of action, direction, and intensity of resultant pressures on walls and bins; and also contains results of tests.

Since it is possible with this apparatus to determine all the elements of a force, the principle involved may also be used in many other ways for laboratory testing, where unknown forces are to be determined.

Tests were made, using river sand and river gravel as fills, the depth of fill being varied from 6 in. to 4 ft. on the retaining wall or weighing gate. The thickness of fill ranges from 6 to 18 in. The fills were also varied by changing the amount of moisture, the method of packing the material, and changing the angle of the surface from horizontal to the angle of repose

It may be seen from the tests that pressure increases with thickness of fill, and that moist or wet material gives a higher pressure than dry material. As would be expected, packed material gives the highest pressure, with the center of pressure in many cases above the center of the retaining surface.

There seems to be no definite relation between the angle of surface and the line of action of the resultant pressure, as deduced by well-known theoretical formulas. Without more exhaustive tests, the author would not attempt to reduce these experimental results to the form of formulas for designing. The tests were started last year and are still being carried on, and it is hoped that something more definite may be reported in the future.

For a proposed design, an excellent method would be to test out a sample of the fill in an apparatus similar to the one described in this paper, with the retaining surface arranged as the proposed wall or bin is to be built. From data thus obtained, the structure could be more intelligently designed.

A LABORATORY METHOD OF DETERMINING PRESSURES ON WALLS AND BINS.

By J. Hammond Smith.

Since there is yet a great deal unknown concerning pressures on walls and bins, due to the action of the retained material, and since great variation exists between theoretical deductions and results in practice, a series of experiments, with special apparatus, has been started in the laboratories of the University of Pittsburgh.

As the title of this paper indicates, the subject is divided into two general classes of structures, walls and bins, yet the experimental results obtained are generally applicable to both classes.

The most important under the class of walls is the common type of masonry retaining wall, supported entirely from the foundation. The back of the wall, which is in contact with the fill, may vary from a smooth vertical, or inclined surface, to one of stepped or irregular form. The condition of the back of the wall, or as we may call it, the actual retaining surface, must of course be a governing factor in the design of apparatus for the determination of pressures upon the wall, due to fill. The rigidity of the wall in relation to its fill is another important factor which must be reckoned with in laboratory testing.

The retaining surfaces of bins are generally supported by attachment around the edges, and frequently by reinforcing members at certain sections and around the top. The variety and relative positions of retaining surfaces of bins are practically unlimited, but the vertical plane, and the plane inclined outward at the top are the most common.

In designing walls and bins, it is important that we should know three things concerning the pressure on a given surface. These are as follows:

1. The location of the center or resultant of pressure on the retaining surface;
2. The direction of the resultant pressure;
3. The intensity of the resultant pressure.

These factors will, of course, have great variation, due to the kind and condition of material to be retained, as well as the method of placing it within or behind the retaining surface. Their values will also depend upon the character of the retaining surface.

With a view of determining the factors, as above mentioned, a special machine was designed and built. The main principle of the machine is that of determining moments of the pressure

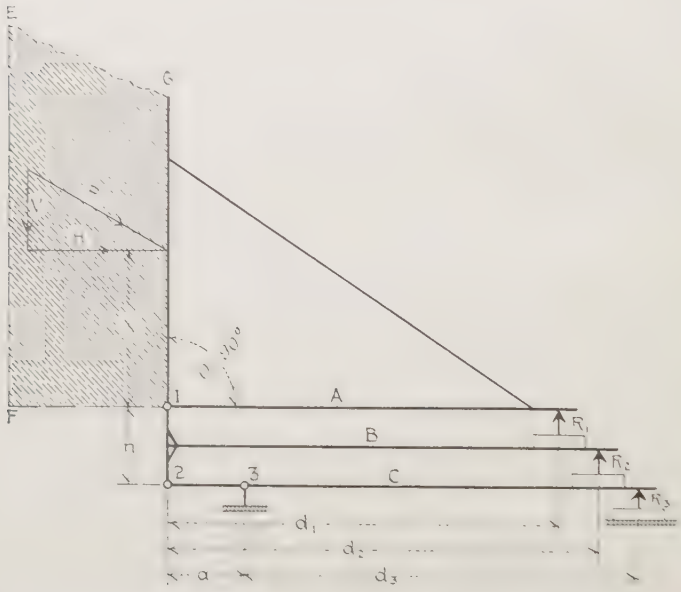


FIG. 1.—Diagrammatic Side View of Apparatus; Retaining Surface Vertical.

on the retaining surface about three different axes. A mathematical discussion of this principle may be drawn from Fig. 1, which is a diagrammatic side view of the apparatus. In this assumption, a short section of vertical wall, or retaining surface, is represented by the line *G*1. The fill is shown in outline, *G*1*FE*. *G*1 is rigidly connected to the arm *A*, and by moment around axis 1, transmits a vertical stress to the weighing scale at R_1, which rests on the arm *B*. The frame, hinged on the axes 1 and 2, is rigidly connected to arm *B*, so that all of the moment

around axis 2 can be determined from the readings of the scale at R_2, which in turn rests on the lower arm C. In like manner, moments around axis 3, which bears upon the main frame of the machine, are determined from the readings of scale at R_3, which rests directly upon the main frame. X is the height of the center of pressure above axis 1. V and H represent the vertical and horizontal components, respectively, of the resultant pressure R. The distance between axes 1 and 2, which lie in the same vertical plane, is represented by n, and a represents the distace between axes 2 and 3, which lie in the same horizontal plane. All of the three axes are horizontal, and therefore should be considered as being normal to the plane of the drawing. The lengths of arm which transmit the weighed vertical forces R_1, R_2 and R_3, are d_1, d_2 and d_3, respectively.

Writing the three moment equations we have:

Around axis 1, $R_1d_1 = HX$ ..... (1)
Around axis 2, $R_2d_2 = H(X+n)$ ..... (2)
Around axis 3, $R_3d_3 = H(X+n) - Va$ ..... (3)

Subtracting equation (1) from equation (2) and solving for H.

$$R_2d_2 - R_1d_1 = Hn$$

$$H = \frac{R_2d_2 - R_1d_1}{n} \quad \ldots\ldots (4)$$

Subtracting equation (3) from equation (2) and solving for V,

$$R_2d_2 - R_3d_3 = Va$$

$$V = \frac{R_2d_2 - R_3d_3}{a} \quad \ldots\ldots (5)$$

Substituting for H in equation (1) and solving for X,

$$X = \frac{R_1d_1n}{R_2d_2 - R_1d_1} \quad \ldots\ldots (6)$$

Referring now to Fig. 2, which shows a modification of the apparatus for measuring the pressures on a retaining surface inclined at an angle θ with the horizontal.—other conditions

being the same as in Fig. 1,—and taking moments as before, we have:

Around axis 1, $R_1d_1 = HX$ ..... (7)

Around axis 2, $R_2d_2 = H(X+n \sin\theta) - Vn \cos\theta$ ..... (8)

Around axis 3, $R_3d_3 = H(X+n \sin\theta - a \cos\theta) - V(n \cos\theta + a \sin\theta)$ ..... (9)

Subtracting equation (9) from equation (8),

$R_2d_2 - R_3d_3 = Va \sin\theta + Ha \cos\theta$ .....(10)

Subtracting equation (8) from equation (7),

$R_1d_1 - R_2d_2 = Vn \cos\theta - Hn \sin\theta$ ..... (11)

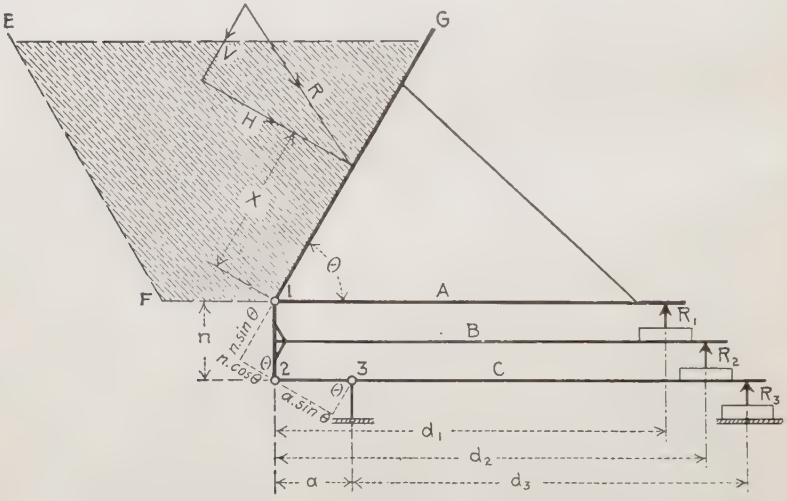


FIG. 2.—Diagrammatic Side View of Apparatus; Retaining Surface Inclined.

Multiplying equation (10) by $\frac{\cos\theta}{a}$,

$$\frac{(R_2d_2 - R_3d_3)\cos\theta}{a} = V \sin\theta \cos\theta + H \cos^2\theta \quad \ldots.(12)$$

Multiplying equation (11) by $\frac{\sin\theta}{n}$,

$$\frac{(R_1d_1 - R_2d_2)\sin\theta}{n} = V \sin\theta \cos\theta - H \sin^2\theta \ldots\ldots(13)$$

Subtracting equation (13) from equation (12), and solving for H.

$$H = \frac{(R_2d_2 - R_3d_3)\cos\theta}{a} - \frac{(R_1d_1 - R_2d_2)\sin\theta}{n} \quad \ldots\ldots (14)$$

Multiplying equation (10) by $\frac{\sin\theta}{a}$,

$$\frac{(R_2d_2 - R_3d_3)\sin\theta}{a} = V\sin^2\theta + H\sin\theta\cos\theta \quad \ldots\ldots (15)$$

Multiplying equation (11) by $\frac{\cos\theta}{n}$,

$$\frac{(R_1d_1 - R_2d_2)\cos\theta}{n} = V\cos^2\theta - H\sin\theta\cos\theta \quad \ldots\ldots (16)$$

Adding equation (15) and equation (16) and solving for V,

$$V = \frac{(R_2d_2 - R_3d_3)\sin\theta}{a} + \frac{(R_1d_1 - R_2d_2)\cos\theta}{n} \quad \ldots\ldots (17)$$

From equation (7), $X = \frac{R_1d_1}{H}$

Substituting for H from equation (14),

$$X = \frac{R_1d_1}{\frac{(R_2d_2 - R_3d_3)\cos\theta}{a} - \frac{(R_1d_1 - R_2d_2)\sin\theta}{n}} \quad \ldots\ldots (18)$$

Equations (14), (17) and (22) reduce to equations (4), (5) and (6), respectively, when the angle θ is made equal to 90 deg.

By use of the above equations, H, V, and X may readily be determined, when R_1, R_2 and R_3 have been measured by experiment.

Fig. 3 shows three sectional views of the apparatus as designed and built for practical laboratory tests. The weighing gate, or front retaining surface, has a width of 18 in., and a height, represented by $G1$, of 4 ft. The weighing gate occupies only the central portion of the front face of the bin; the adjustable ledges at the sides of the gate being attached to the main frame. The weighing gate, as may be seen from the elevation and plan of Fig. 3, has two walls; the inner wall forms the retaining surface, while the outer wall is intended to catch

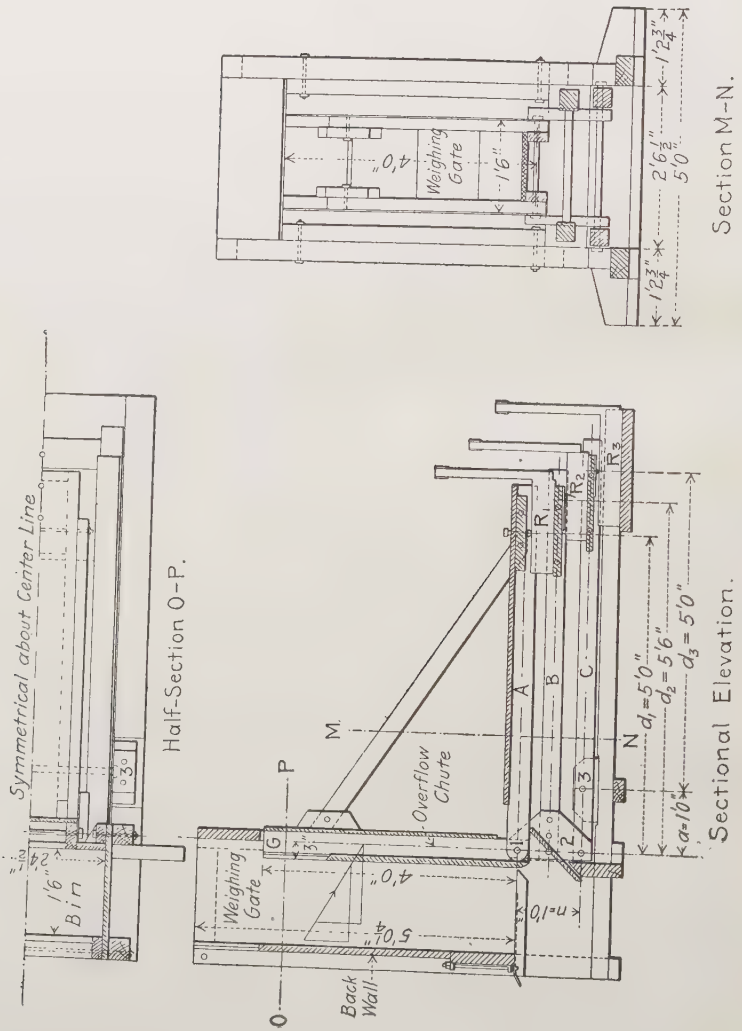


FIG. 3.—Sectional Views of Apparatus.

any of the filling material which may run over the inner wall, and return it to the space beneath the bin. The height of the inner wall is adjustable by means of removable sections. Therefore a fill of given depth, with its angle of repose, may be obtained readily by filling the bin until the excess material runs over the inner wall of the gate. The clearance space between the gate and side ledges and bottom is covered with loosely fitting strips of muslin, glued to the inner surface of the

FIG. 4.—Apparatus for Determination of Pressures on Walls and Bins.

bin. The bin has vertical side and back walls, is 28½ in. wide inside, and 5 ft. high above its floor. The back-wall is made adjustable, so that thickness of fill between back-wall and weighing gate may be varied. This ranges from 6 to 18 in., when the weighing gate is in a vertical position. The back-wall is suspended on an adjustable rod at the top, which allows it to swing outward at the bottom when the fill is to be discharged from the bin. It also has removable sections to facilitate the filling of the bin. All inner surfaces of the bin were smooth surfaced by planing, and finished with one coat of paint.

TABLE I.—PRESSURES ON WALLS AND BINS; WEIGHING GATE VERTICAL.

MATERIAL, WET GRAVEL, 118 LB. PER CU. FT.; THICKNESS OF FILL, 6 IN.; ANGLE OF SURFACE, 40 DEG. (ANGLE OF REPOSE).

Test No.	Depth of Fill on Weighing Gate, ft.	R_1, lb.	R_2, lb.	R_3, lb.	H, lb.	V, lb.	X, ft.	Resultant of Total Pressure on Weighing Gate, lb.
1	1	4.06	11.56	9.75	43.28	14.83	0.56	45.80
	2	16.81	31.75	26.05	90.56	44.32	1.05	101.00
	3	33.96	54.00	45.75	127.20	68.25	1.47	144.70
	4	56.81	81.25	68.50	162.82	104.37	1.90	193.20
2	1	4.50	12.56	10.25	46.59	17.84	0.57	49.60
	2	19.69	35.66	27.81	97.67	57.06	1.15	113.00
	3	39.72	63.28	53.37	149.45	81.19	1.45	170.00
	4	57.75	83.19	69.80	169.15	108.53	1.88	201.00
3	1	4.50	12.56	11.00	46.59	14.09	0.56	48.50
	2	19.44	36.25	30.00	102.19	49.37	1.05	113.60
	3	38.16	60.75	52.00	144.12	74.12	1.45	162.00
	4	59.00	84.50	72.00	169.75	104.75	1.90	199.00
Aver.	1	.....	.....	.....	45.49	15.59	0.56	47.96
	2	.....	.....	.....	96.81	50.25	1.09	109.20
	3	.....	.....	.....	140.26	74.52	1.45	158.90
	4	.....	.....	.....	167.24	105.88	1.89	197.70
MATERIAL, RIVER GRAVEL, SLIGHTLY MOIST, PACKED, 113 LB. PER CU. FT.; THICKNESS OF FILL, 10 IN.; ANGLE OF SURFACE, 0 DEG.								
1	1	7.25	22.07	25.00	85.14	−3.62	0.41	85.40
	2	30.75	55.69	55.79	152.55	27.25	1.05	155.00
	3	72.37	106.00	106.25	221.13	51.75	1.70	227.00
	4	110.07	146.00	143.50	251.65	85.50	2.27	265.30
2	1	5.47	16.88	18.50	65.46	0.30	0.42	65.47
	2	33.47	62.25	66.94	175.03	7.71	0.96	175.20
	3	70.06	103.88	103.44	221.00	54.13	1.65	227.20
	4	121.03	159.50	160.02	272.10	77.25	2.28	283.20
3	1	7.06	20.50	24.25	77.45	−8.50	0.43	77.90
	2	31.88	57.75	60.37	158.25	15.75	1.03	159.50
	3	61.13	90.00	90.00	189.38	45.00	1.68	194.50
	4	100.00	133.50	130.00	234.25	84.25	2.23	249.00
4	1	8.13	24.31	29.37	93.09	−13.54	0.40	94.00
	2	26.00	50.87	49.13	149.81	34.18	0.93	153.80
	3	63.19	93.87	92.87	200.38	51.93	1.65	207.00
	4	101.77	134.37	132.00	230.19	79.06	2.30	243.50
Aver.	1	.....	.....	.....	80.28	−6.24	0.41	80.69
	2	.....	.....	.....	158.92	21.22	1.00	160.82
	3	.....	.....	.....	208.00	50.90	1.67	213.90
	4	.....	.....	.....	247.05	81.51	2.27	260.10
MATERIAL, DRY SAND, LOOSE, 90 LB. PER CU. FT.; THICKNESS OF FILL, 10 IN.; ANGLE OF SURFACE, 0 DEG.								
1	1	2.25	7.32	3.50	29.01	22.76	0.56	36.90
	2	11.37	22.31	13.50	65.85	55.20	1.07	85.80
	3	29.81	47.75	35.50	113.57	85.10	1.50	141.50
	4	48.88	70.63	54.25	144.06	117.18	1.90	186.00
2	1	2.56	8.62	6.57	34.61	14.56	0.48	37.50
	2	12.12	26.37	20.50	84.18	42.28	0.83	94.00
	3	28.56	48.27	37.20	122.61	79.49	1.32	146.00
	4	50.25	73.27	57.50	151.74	115.49	1.87	191.00
Aver.	1	.....	.....	.....	31.81	18.66	0.52	37.20
	2	.....	.....	.....	75.00	48.74	0.95	89.90
	3	.....	.....	.....	118.59	82.29	1.41	143.70
	4	.....	.....	.....	147.84	116.33	1.88	188.50

TABLE I.—(*Continued.*)

MATERIAL, SAND SLIGHTLY MOIST, PACKED, 91 LB. PER CU. FT.; THICKNESS OF FILL, 10 IN.; ANGLE OF SURFACE, 0 DEG.

Test No.	Depth of Fill in Weighing Gate, ft.	R_1, lb.	R_2, lb.	R_3, lb.	H, lb.	V, lb.	X, ft.	Resultant of Total Pressure on Weighing Gate, ft.
1	½	1.88	7.76	9.37	33.42	−4.05	0.25	33.60
	1	6.44	18.69	20.63	70.56	−0.36	0.46	70.60
	1½	13.56	29.13	28.50	92.38	17.68	0.78	93.50
	2	24.00	43.00	41.00	116.50	31.50	1.10	121.00
2	½	1.50	5.94	6.88	25.10	−2.20	0.28	26.04
	1	6.00	17.81	19.37	68.00	1.15	0.44	68.20
	1½	13.75	30.13	30.00	96.93	15.68	0.75	98.00
	2	24.13	43.00	41.63	115.90	28.40	1.10	119.20
Aver.	½	.....	.....	.....	29.26	−3.13	0.27	29.82
	1	.....	.....	.....	69.28	0.39	0.45	69.40
	1½	.....	.....	.....	94.66	16.68	0.76	95.75
	2	.....	.....	.....	116.20	29.90	1.10	120.10

TABLE II.—PRESSURES ON WALLS AND BINS; WEIGHING GATE VERTICAL.

AVERAGE RESULTS.

Material.	How Placed.	Number of Tests.	Angle of Surface, deg.	Thickness of Fill, in.	Depth of Fill on Weighing Gate, ft.	H, lb.	V, lb.	X, ft.	Resultant of Total Pressure on Weighing Gate, lb.
Moist gravel, 115 lb. per cu. ft.	Loose	8	0	6	1	31.58	9.47	0.43	33.07
					2	72.21	31.20	0.86	78.00
					3	117.27	56.14	1.35	130.10
					4	156.76	80.10	1.70	175.10
Dry gravel, 113 lb. per cu. ft.	Packed	2	0	6	1	101.14	−3.81	0.43	101.40
					2	163.22	26.65	1.05	164.90
					3	197.36	51.59	1.75	203.20
					4	229.18	78.87	2.38	240.60
Dry gravel, 113 lb. per cu. ft.	Loose	8	0	10	1	38.40	5.75	0.42	34.00
					2	87.04	22.11	0.84	90.00
					3	134.48	56.00	1.20	149.10
					4	186.13	90.18	1.60	206.00
Dry gravel, 113 lb. per cu. ft.	Loose	5	0	14	1	38.35	4.69	0.41	39.36
					2	82.43	23.26	0.84	85.74
					3	130.59	55.62	1.19	141.14
					4	183.64	87.56	1.61	202.28
Dry gravel, 113 lb. per cu. ft.	Loose	4	0	18	1	38.01	8.13	0.42	38.99
					2	92.70	31.14	0.83	97.47
					3	139.78	64.39	1.17	154.19
					4	193.64	98.48	1.60	219.47
Dry sand, 90 lb. per cu. ft.	Loose	2	0	6	1	32.50	11.87	0.42	32.50
					2	73.76	43.26	0.91	85.50
					3	98.85	82.00	1.35	128.00
					4	128.42	99.22	1.69	162.00
Moist sand, 94 lb. per cu. ft.	Packed	2	0	6	1	76.00	7.00	0.52	76.25
					2	154.00	19.25	1.14	155.20
					3	171.00	38.00	1.87	175.40
					4	183.00	59.00	2.56	187.50
Moist sand, 94 lb. per cu. ft.	Loose	4	0	10	1	24.14	7.16	0.31	25.30
					2	63.25	33.22	0.63	71.40
					3	105.45	73.00	1.05	121.00
					4	161.60	91.71	1.55	182.60

The distances a and n are each equal to 1 ft., while the arms of the weighed forces are $d_1 = 5$ ft., $d_2 = 5$ ft. 6 in., and $d_3 = 5$ft. (See Fig. 3).

As it was not practicable to use knife-edge bearings for axes 1, 2, and 3, $1\frac{1}{4}$-in. cold-rolled shafting with heavy ball bearings at each end was used. There was some question as to the friction and exact position of the center in the ball bearings used. Therefore, a special device was constructed for the testing of these bearings. The results showed that the centers

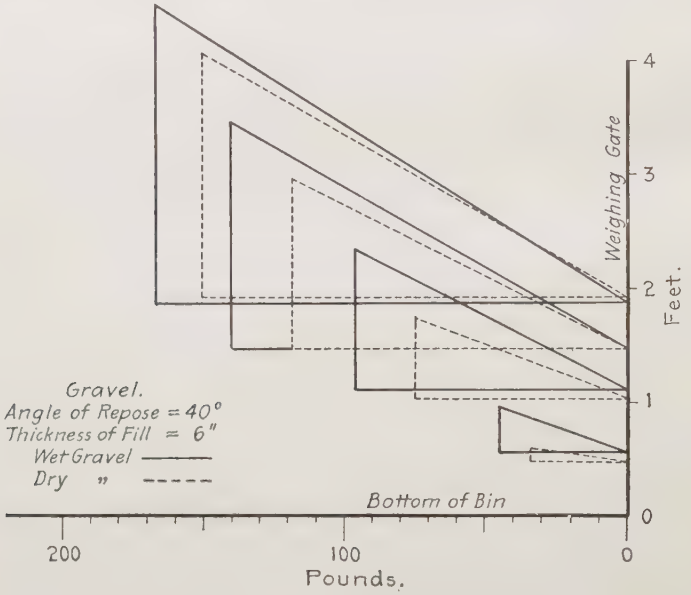


FIG. 5.—Graphical Representation of Pressures—Gravel.

of the bearings were quite true, and that they were practically frictionless, even under heavy loads.

All framework of the apparatus is very heavy, and designed for rigidity. The movement of the scale beams is limited to a small amount, so that the movement of the weighing gate will be the least possible. Fig. 4 shows a side view of the apparatus ready for testing.

In Tables I and II will be found results from tests made with fills of river gravel and river sand; the weighing gate or

retaining surface being in a vertical position during these tests. The thickness of fill, or normal distance between weighing gate and back-wall, varied in this set of tests from 6 to 18 in. The distance represented by $G1$ in Fig. 1, corresponds to the column in Tables I and II headed "depth of fill on the weighing gate in feet." The moment forces in pounds are given under headings R_1, R_2 and R_3 (in Table I), from which the horizontal component H, vertical component V, and resultant of total pressure on the weighing gate are determined in pounds by use of equations

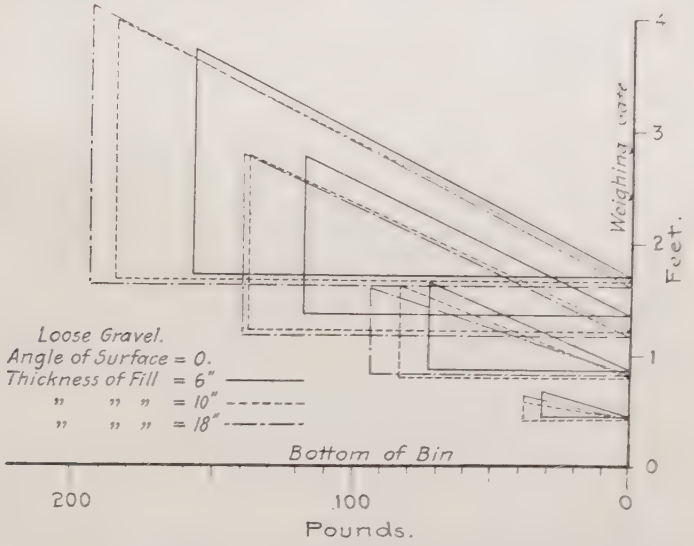


FIG. 6.—Graphical Representation of Pressures—Loose Gravel.

(4) and (5). X, the height in feet of center of pressure above the bottom of fill, is determined by use of equation (6).

When the values of a, d and n, used in our apparatus, are substituted these equations reduce to:

$$H = 5\ (1.1\ R_2 - R_1) \quad \ldots\ldots\ldots\

The weighing surface being set $\frac{1}{4}$ ft. back of the vertical plane through axis 1 brings the additional term, $V/4H$, into the value of equation (6) for X.

Table II shows average results for various conditions and various thicknesses of fill. The vertical component V was frequently found to be negative, or acting upward, for packed fills of 6 in. and 1 ft. in depth. This was probably due to an inverted arching action.

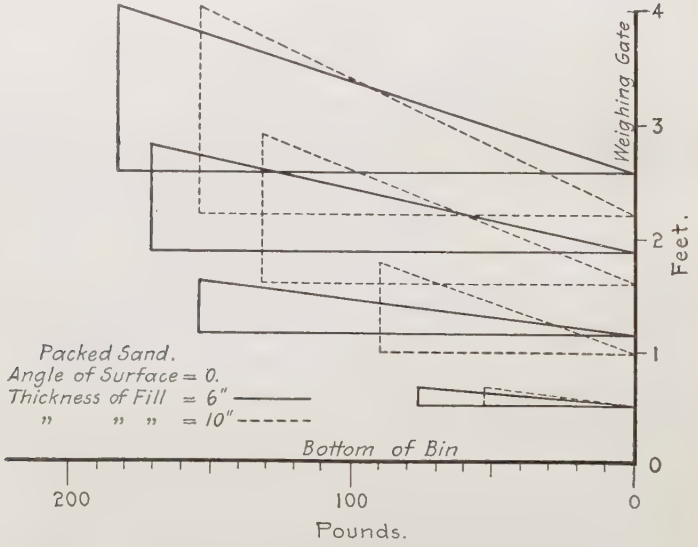


FIG. 7.—Graphical Representation of Pressures—Packed Sand.

Figs. 5, 6 and 7 show the pressures graphically with their relative positions and amounts for depths of fill on weighing gate of 1, 2, 3 and 4 ft. The right-hand vertical line represents the weighing gate, and the lower horizontal line the bottom of the bin.

The author wishes to acknowledge his great appreciation of the assistance rendered in carrying on tests by the following students of the University of Pittsburgh: Messrs. W. E. Kaye, W. P. Hall, W. H. Virgin and B. Goldberg.

DISCUSSION.

Mr. G. L. Fowler.—In working along similar lines, I recently made some investigations, using pipe closed at one end with a diaphragm of cheap wood-pulp writing paper. With a 6-in. pipe and 21 in. of sand over the paper, it took about 2800 lb. applied to a plunger at the top of the sand to burst the diaphragm. With a 4-in. pipe and 16 in. of sand, it took 4875 lb. and with a 2-in. pipe and 9 in. of sand, it took 10,000 lb. In a box now in use, I have vertical floating planks arranged at different heights or depths to measure the lateral thrust. They are held in place by platform scale levers bearing on the center of a diaphragm dynamometer. This is very sensitive and registers the pressure with no appreciable movement of the plank. Mr. Fowler.

In all of these experiments, it will be understood that I am working with a superimposed load, ranging from the mere weight of the sand to 300 tons. The author, I understand, has not experimented with superimposed loads.

It seems, also, that the weighing gate was too large to show delicate proportions of load to increasing depth.

Mr. Alan E. Flowers.—I wish to ask the author of the paper whether he took any precautions to jar the apparatus so as to bring the material to its final angle of repose, and if not, whether that would not be a desirable thing to do in order to cause the loose materials to come to a final resting place, and possibly therefore bring about different conditions of pressure than those actually measured. Mr. Flowers.

Mr. Edgar Marburg.—I should like to say that on reading this paper in manuscript, the first impression I gained was that it struck a distinctly new note. I think that the author of the paper, who designed this apparatus, deserves generous recognition for having gotten up something simple and convincingly accurate in principle, for determining exact values concerning phenomena of great complexity. So far as I know, the apparatus described Mr. Marburg.

Mr. Marburg. in this paper marks a distinctly new departure and opens up large possibilities for investigation and inquiry into obscure phenomena, including the determination in a quantitative sense of the differences between the conjugate pressure and the abutting intensity of materials of various kinds, the latter being, as is well known, theoretically very much higher than the former.

The determination of these values in a comparative way is of practical interest in the design of foundations, arches, etc., and I should like to ask the author whether such determinations have been made or contemplated.

The Chairman. THE CHAIRMAN (PRESIDENT A. W. GIBBS).—The Chair would like to add two questions to those of Mr. Marburg. First, was there any evidence of slip? The gate only occupies part of the face, and I should like to know whether there is any yielding in the form of cleavage and slip. The other question is whether the author has made or intends to make any tests to show the effect of a rolling—that is, traveling—load on the top in choosing the height of the center of pressure, X. Where trains are running on the top of an embankment or retaining wall, would not that tend to elevate the center of pressure?

Mr. Olshausen. MR. G. R. OLSHAUSEN.—I should like to ask in this connection whether experiments were undertaken to determine the lateral pressure in grain elevators. There we encounter special conditions, in that we have to consider the energy of the moving mass in addition to the static pressure.

Mr. Smith. MR. J. HAMMOND SMITH.—In answer to Mr. Fowler's question, no experiments have been made upon cases where we had a superimposed load, that is, a load above what would be obtained by the angle of repose. Now in regard to the weighing gate, of course, this weighing gate does not occupy the whole of the opening on one side, but it is confined simply to the center. There are two ledges, one on each side, which are flush with the weighing gate itself; the clearance openings between the two being very carefully covered with loose strips of canvas in order to keep the material from getting in and causing friction. Of course, the proximity of the weighing gate to other surfaces was taken into account when the apparatus was designed. We wanted to be sure that we had the weighing gate small enough in relation to the size of the bin. There may be some question

as to whether it was small enough in this case. It seems that Mr. Fowler understood that the pressure on the bottom was weighed as well as the pressure on the side of the bin. This apparatus weighs only the pressure on the one plane surface on the side of the bin. Of course, by connecting the bottom with the side of the bin, an arrangement could be made by which the pressure could be weighed, not only on the side, but also on the bottom. The shape of that surface could be varied by simply having the bottom or back of the wall shaped as might be desired. The idea would be to have that shape similar to the shape in the proposed design of the wall or bin, in testing it out for a particular purpose. Mr. Smith.

In answer to Mr. Flowers' question as to the final angle of repose, the material was introduced without any jar, that is, in the case of the angle of repose. The bin was filled until the material ran over the inside part of the weighing gate. That we consider to be the angle of repose. Of course, by jarring the apparatus, we would probably have gotten a somewhat different angle of repose, but it does not seem to me that it would have been very much different; also by jarring the apparatus, we would have a tendency to pack the material, which would not be natural in a good many cases.

In regard to Mr. Marburg's question as to determination of the abutting intensity by applying reverse pressure, that is, by filling the bin and then putting reverse pressure upon the weighing gate by an inward movement, we have done very little along that line; but we have made one or two experiments in which we found that when we attempt to reverse the pressure on the wall, the resultant pressure increases very rapidly, although I have no data to show just what it is.

Answering Mr. Gibbs' question about slips; we have made this apparatus just as rigid as possible, the idea being that the weighing gate should not move, or if it moved at all, it should move an extremely small amount in relation to the bin, because as soon as movement takes place, the pressure will decrease and therefore the moment of the weighing gate b comes very small. The scale beams were limited in their movement to the least possible amount so that there would be very little movement of

Mr. Smith. the gate; and we are quite sure that there is no appreciable slip due to any movement or yielding of the gate.

In regard to the pressure of grain in the static condition, or in motion, we have made no attempt at studies of that kind, but I suppose this apparatus or its principles could be used in such investigations.

A CYLINDER FRICTION AND LUBRICATION TESTING APPARATUS.

By Alan E. Flowers.

SUMMARY.

An apparatus is described which is capable of testing cylinder lubricants at various rates of feed under piston-ring pressures from 2 to 9 lb. per sq. in. and at steam temperatures up to 800° F. ($426\frac{2}{3}$° C.).

The cylinder has an 8-in. bore, the stroke is 8 in., and at present the apparatus is being run at speeds up to 300 r.p.m.

The results of some tests are plotted showing the actual readings as taken and the effects of steam pressure, steam temperature, and cylinder-wall temperature with and without steam, on the friction coefficient for two different lubricants.

The contents of the paper are further summarized in the eleven conclusions at the close of the paper.

A CYLINDER FRICTION AND LUBRICATION TESTING APPARATUS.

By Alan E. Flowers.

Introduction.

The work on this subject was begun at the University of Missouri in 1909 and a small apparatus embodying the principle was constructed and tried out.[1]

With the information obtained from the small apparatus, a much larger apparatus was designed and has been assembled at the Ohio State University, where it is now set up and available for tests to determine the characteristics of cylinder friction and for the comparison of different cylinder lubricants under the various conditions of steam temperature, steam pressure, piston-ring pressure, speed and oil feed that obtain in service.

Principle of the Apparatus.—The apparatus consists of a steam cylinder within which two pistons connected by a piston rod are placed. The space between the two pistons has a constant volume so that steam admitted to this space does no work on the pistons. Two steam pipes are brought into the cylinder at points just short of the parts rubbed over by the piston so that oil carried along by the steam may readily reach the rubbed surfaces.

An electric motor drives the two pistons by means of a crank disk and connecting rod, and the net input of the motor is a direct measure of the friction of the two pistons and piston rings. Steam is supplied by a gas burning coiled-steel-tube boiler.

Fig. 1 shows a diagrammatic sketch illustrating the main principle of the apparatus, and in Fig. 2 is given a photograph showing the general arrangement.

Details of the Construction Employed.

Boiler.—The boiler, manufactured by the White Company, is made of $\frac{3}{8}$-in. coiled steel tube, capable of working at pressures

[1] This apparatus was described in the University of Missouri Bulletin, Engineering Experiment Station Series, Volume 2, No. 2, June, 1911

up to 1000 lb. per sq. in. and at temperatures up to 800° F. (426⅔° C.). Natural gas and compressed air are supplied to the burner, which is capable of giving considerable degrees of superheat.

Cylinder.—The cylinder is 8 in. in bore and 36 in. long. A 3-in. drain leads out from the middle of the bottom. The cylinder and first set of piston rings were manufactured by McIntosh, Seymour & Co.

Connecting Rod.—The connecting rod is 33 in. long and the stroke is 8 in. This makes the maximum angularity of the con-

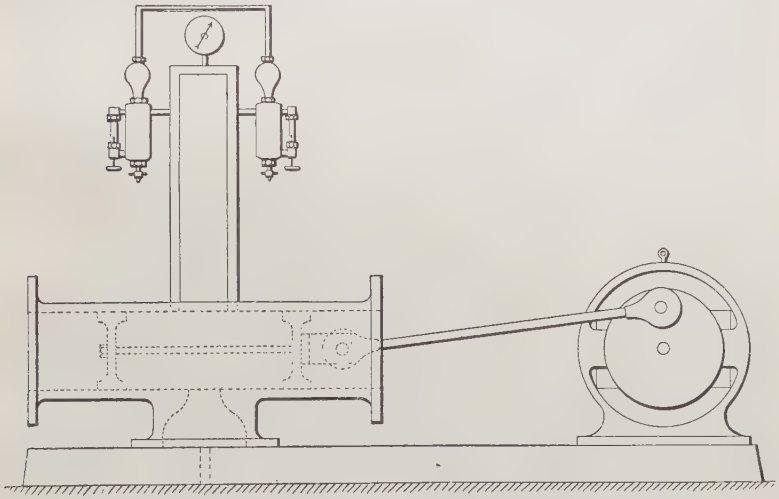


FIG. 1.—Diagram Illustrating Principle of Cylinder Friction Apparatus.

necting rod less than 7 deg., a value so small that its effect is negligible. Hess-Bright ball bearings at each end of the rod make the connecting rod friction negligible compared to the cylinder friction.

Motor.—A General Electric 110-volt, 400-1200 r.p.m. motor with two identical armature windings and commutators, each rated at 5.5 amperes drives the pistons. One of these armature windings is used as a motor winding and the other is used to give the generated electromotive force. For any constant value of field excitation, the spare winding also gives an electromotive force which is proportional to the speed. Suitable resistances

can be connected in series with a voltmeter so that the speed can be read directly on a suitable scale and so can easily be kept constant. The motor is provided with interpoles and shunt-field speed adjustment, but is being run at present with constant field and variable applied voltage. A motor generator set supplies the power to drive the motor, and rheostats in the separately excited generator field circuit make it a simple matter to control the voltage of the generator, so adjusting

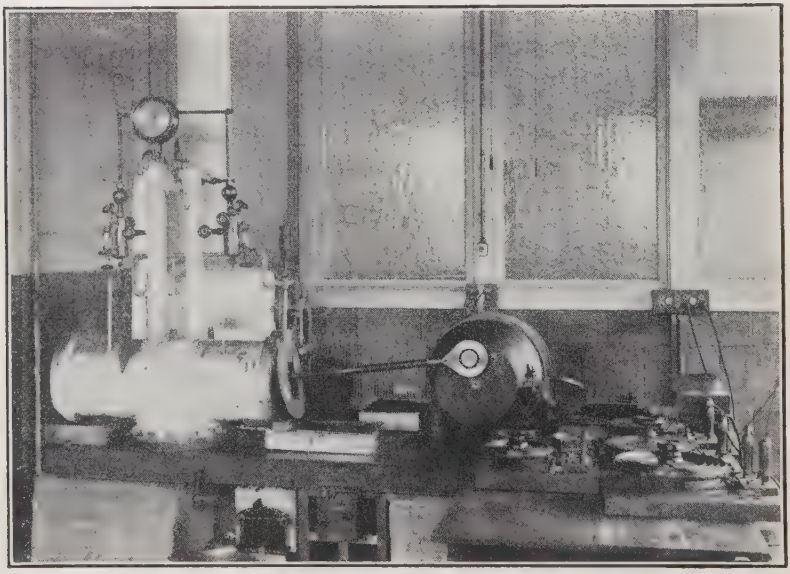


FIG. 2.—Apparatus for Testing Cylinder Lubricants.

or keeping constant the speed of the motor driving the pistons. The motor is fitted with ball bearings and was specially designed for this service, in order to have the lowest possible no-load losses.

Oil Feed.—Two 1-pint Detroit sight-feed lubricators supply oil in the usual manner to the steam pipes.

Fuel.—Natural gas and compressed air are supplied and are controlled by hand-operated valves, which can be adjusted to give the desired combination of steam pressure and superheat temperature.

Water.—A double-plunger pump driven by an adjustable speed motor controls the water supplied to the boiler. The water supply is taken directly from the city water mains which supply water at a pressure of approximately 65 lb. per sq. in. A hand-operated valve between the water mains and the pump provides an additional control for the water.

Piston Rings.—The piston rings at present in use are of the constant radial thickness type. Each piston has five piston-ring slots and one or all five may have piston rings placed in them. All the piston rings have a $\frac{3}{8}$-in. face and each of the four sets has a different radial thickness so that the intensity of pressure exerted by the piston rings against the cylinder wall may have values from 2 to 9 lb. per. sq. in.

Steam Temperature and Pressure Measurement.—The boiler has a high-pressure steam gage reading to 1200 lb. per sq. in. An Ashcroft standard test gage, reading to 300 lb. per sq. in., is connected to the steam line just ahead of the steam cylinder. A valve between the boiler and cylinder can be used to throttle or to cut off the steam. At one time a pressure-reducing valve was also placed between the boiler and cylinder, but owing to the high degree of superheat to which it was subjected, it did not operate satisfactorily and was removed

A thermometer pocket is placed in the steam line near the boiler, another in the center of the cylinder steam-space and two in the walls of the cylinder, one at each end and opposite the midstroke position of the pistons.

The cylinder-wall pockets were drilled very close to the inner wall of the cylinder so that the thermometer in them would give the average working temperature of the friction surfaces.

High-grade thermometers guaranteed accurate to one-half a division, manufactured by the Hohmann & Maurer Division of the Taylor Instrument Co., are used to read the temperatures.

PROPOSED MODIFICATIONS.

The cylinder ends were provided with flanges so that the ends could be covered and the exhaust side of the pistons subjected to any desired steam pressure. When the ends are not covered the exhaust side of the pistons is subjected to atmospheric pressure and the corresponding steam temperature by the steam that

leaks past the rings. Thus the rubbed surfaces are alternately exposed to the differences of temperature and pressure that obtain in service when running non-condensing. The difference in pressure on the two sides of the piston is an important element in the amount of oil feed necessary, and the difference of temperature; an important element in determining the viscosity and therefore the amount of friction encountered by the pistons and piston rings.

In order to further increase the severity of tests with high degrees of superheat, an electric heating coil has been designed which is to be placed around the cylinder. This will produce a heat flow inwards and raise the wall temperature, as well as to tend to raise the steam temperature.

The sight-feed lubricator gives a rate of flow for any setting of its control valve which is determined by the viscosity of the oil. As the control valve is not near the steam pipes, it is not much above the temperature of the room. At these temperatures the viscosity of cylinder lubricants is very great and changes enormously with small changes of temperature. This makes it almost impossible to get a high rate of oil feed or to keep the rate of oil feed constant. Moreover, the size of the drop varies with the temperature, the steam pressure and with the rapidity with which the oil is fed, so that the rate of oil feed in drops per minute bears no definite ratio to the pints per hour or any other definite measure.

Oils differ widely in the size of the drops they will form, owing to their variations in surface tension, so that different oils fed at the same number of drops per minute do not necessarily supply the same volume of oil to the rubbing surfaces.

An oil pump working without slip which will feed any oil at a constant rate which can easily be predetermined or adjusted, is being designed for this service.

RESULTS OF TESTS.

A graphical log of a test is plotted in Fig. 3 to show a set of readings as taken and their variations. The value of the current required to drive the motor alone is also plotted as the horizontal line at 0.52 amperes. The difference between this no-load current and the total current gives the net current required

for the cylinder friction. For this test the friction coefficient could be obtained by multiplying the net current by the factor 0.01366.

An appreciable part of the variations shown here is due to the difficulty of controlling the oil feed. There was an excessive rise in friction amounting to 300-400 per cent when the steam pressure was raised quickly from atmospheric pressure to 120 lb. per sq. in. This excessive friction does not occur when the steam pressure is increased gradually or when it is decreased to a lower

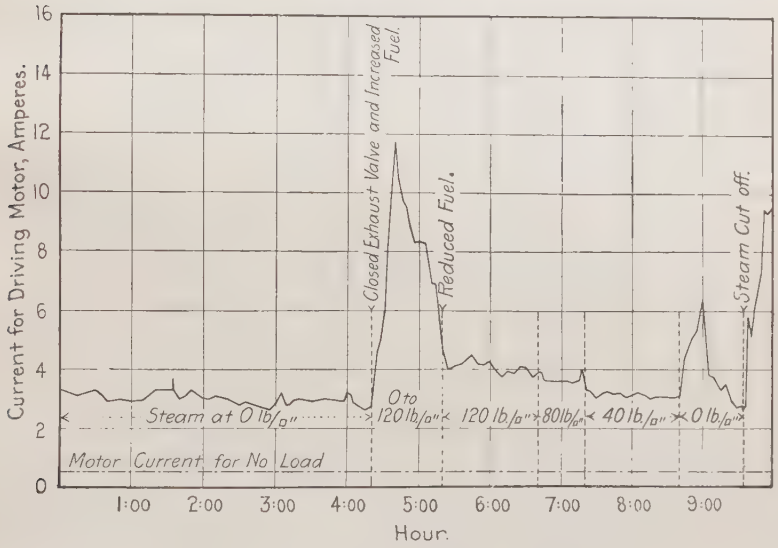


FIG. 3.—Readings as Taken in a Test.

value; it may be due to the sudden blowing of the oil out from the space between the piston rings and the cylinder walls.

The effect of steam pressure on the friction is shown in Fig. 4. Saturated steam was used in these tests, and results are plotted for two different cylinder lubricants. The friction for low-steam pressure is practically the same for the two lubricants, but at the higher pressures one lubricant gives appreciably more friction than the other.

As the viscosity of oils decreases very greatly with increased temperature, the results given in Fig. 4 are replotted in Fig. 5,

but referred to the temperatures measured by the thermometers in the cylinder-wall pockets. Fig. 5 also gives for comparison the friction coefficient for each of the two cylinder lubricants with the steam shut off and the cylinder cooling off. It is noteworthy that these friction curves taken without steam show the increase in friction at lower temperatures that should occur with the correspondingly increased viscosity. It is noteworthy also that the curves show the same relative difference between the two lubricants.

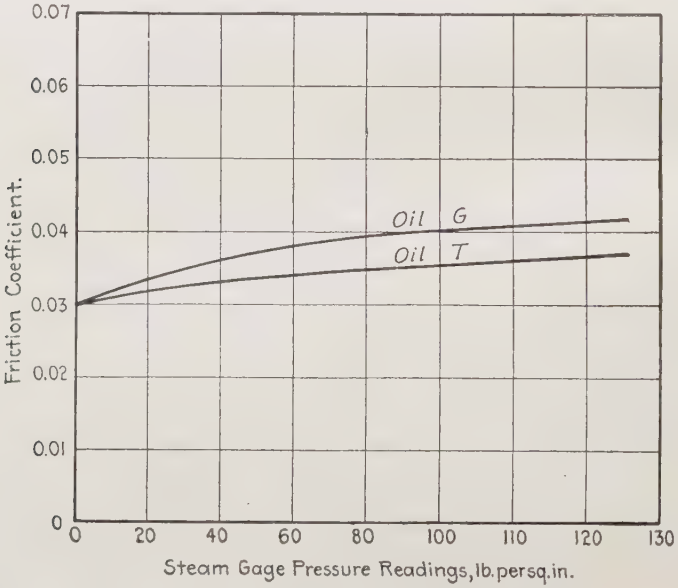


FIG. 4.—Effect of Steam Pressure on Friction.

At 97° C. the friction coefficient for both lubricants is the same, and both show a coefficient more than twice as great as that obtained with saturated steam at atmospheric pressure.

At the higher temperatures, both show a much lower friction when free from steam than when saturated steam of the corresponding pressure is present.

The larger friction coefficients at the highest recorded temperatures are probably not significant of anything more than the transition from the condition with saturated steam present under

pressure, to the condition of atmospheric pressure and the absence of steam or water. It is possible, however, that at the higher temperatures the viscosity of the lubricant may be reduced to a point such that it is readily squeezed out from between the rubbing surfaces, and by bringing the surfaces close together, or even into contact, to increase the friction between them.

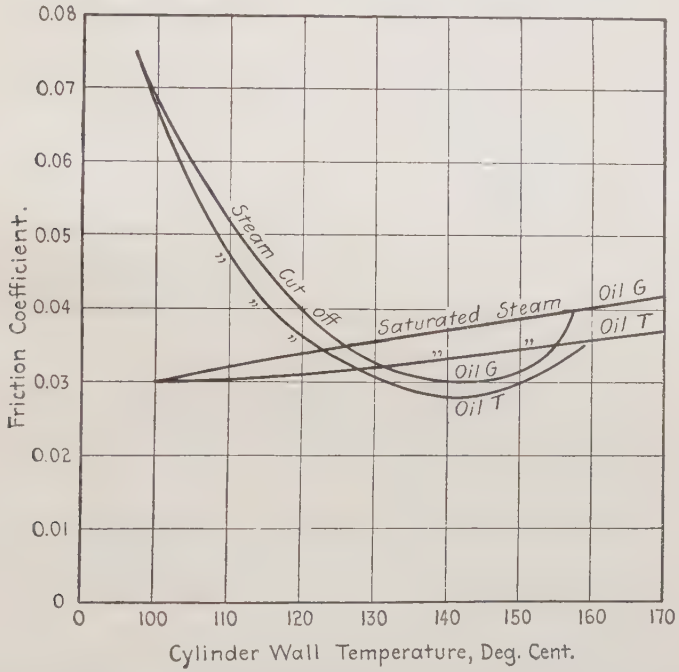


FIG. 5.—Results Shown in Fig. 4 Referred to Cylinder-Wall Temperatures, and Effect of Cooling Cylinders.

Other tests showed that when the feed was scanty, the friction was not only large but subject to sudden large variations.

The effect of shutting off one of the two lubricators was to increase the friction within a few minutes in spite of the fact that some oil was carried to the end at which the lubricator had been shut off. The amount of lubricant carried over in this way to the unlubricated piston was sufficient to keep the surfaces from stick-

ing or cutting, but not sufficient to give the low friction coefficient corresponding to the *total* amount of lubricant fed.

Shutting off all the lubricant increases the friction appreciably within five minutes, when steam is present under pressure, but when steam is present at atmospheric pressure it takes at least 30 minutes for the friction to change. This difference is doubtless due to the carriage of the lubricant to or away from the rubbing surfaces when steam is present under pressure.

CONCLUSIONS.

1. An apparatus has been constructed which will readily measure the friction coefficient between piston or piston rings and cylinder-wall surfaces.

2. The conditions of operation of the rubbing surfaces reproduce closely the conditions of service.

3. Tests may be carried out at various piston-ring pressures, steam pressures, and steam superheat temperatures covering the ranges found in practice.

4. The modifications described will make it possible to impose conditions more severe than those met with in practice.

5. Different lubricants do not give necessarily the same friction coefficient, and their lubricating value can be determined by means of this apparatus.

6. Constancy of friction requires a liberal supply of lubricant, particularly when there is a difference of steam pressure on the two sides of the piston. A large amount of lubricant is carried directly on out with the exhaust.

7. Cutting off or reducing the supply of lubricant brings about an increase of friction within 5 minutes.

8. Increasing the supply of lubricant may bring about a reduction of friction within 15 minutes, but at low pressures, or when starting a run with surfaces free from oil, it may take two hours to reach a consistently low value of friction.

9. An increase in the rate of feed of the lubricant makes a considerable reduction in the friction when the amount fed is small, but with a continued increase of feed of lubricant, the friction tends to approach a certain minimum value.

10. The friction coefficients obtained so far are all large,

3 to 4 per cent or more, and very much larger than the values found in well-lubricated bearings.

11. The friction coefficients obtained and the amount of lubricant necessary are greatly modified by the presence of steam and by the difference of steam pressure on the two sides of the piston.

It is hoped that discussion of the construction and operation of the apparatus may lead to further improvements.

It is a pleasure to acknowledge here assistance in this work from the University of Missouri, Cornell University, the Ohio State University, which has contributed largely for the construction of the apparatus described here, and the Telluride Association, which contributed part of the expenditures for this work. Acknowledgment is also made to Mr. M. P. Weinbach, Prof. C. F. Hirshfeld and Dr. P. H. Conradson for suggestions during the progress of the work.

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DISCUSSION.

Mr. Conradson. MR. P. H. CONRADSON.—I should like to ask Mr. Flowers a few questions in regard to his machine. I have had the pleasure of seeing it in operation, and was very much impressed with the possibility of adapting it to something useful in connection with the testing of lubricants. Mr. Flowers has said that the method of feeding the oil has a great deal to do with the result. He also states that there is a great deal of difference whether this machine is run with or without steam at the same temperature. That is true. I should like to ask him whether he knows what the actual condition of the oil is when it reaches the steam cylinder. Does the oil flow from the lubricator in liquid form, and has any attempt been made to atomize it and to spray it over? It is well known that the cylinder oils are very heavy and sluggish even at high temperatures, and unless the oil is admitted to the steam chest or the cylinder in a very highly atomized form, the results will not be of the best. Therefore, I would suggest as the next step in developing this apparatus, that the method of feeding the oil into the steam cylinder be in a little different form, which would probably lead to different results. I should also like to ask whether the cylinders in which the tests were performed were open or not.

The results would also depend on whether cool air is drawn in. That changes the condition of the lubricant to a great extent, and conditions for test purposes can never be constant unless the steam is admitted on both sides of the piston. In such case the temperature conditions will be fairly uniform, and I think that this would give more valuable results. It is true that in steam cylinders, at the beginning of the stroke, the pressure is highest and the temperature highest, and as the stroke goes down, the pressure gets less and less and the temperature falls, but in ordinary steam cylinders and in locomotive service the inside cylinder surfaces are never exposed to atmospheric air, except in certain classes of crank case engines.

Mr. Conradson.

I should like to ask Mr. Flowers if he has done anything further to develop his apparatus for determining the coefficient of friction by having the cylinders under constant conditions as to pressure, temperature, and feed of oil; that is, if the testing machine has been provided with caps or heads at each end of the steam cylinder to bring about these conditions. If so, much more valuable and interesting practical results would be obtained.

Mr. Flowers.

Mr. Alan E. Flowers.—It is with pleasure that I acknowledge the very great help that has been given in the course of this work by Mr. Conradson, both at this meeting and previously. Discussion here or contributed later will be welcomed, because in a research of this character, which is an attempt to duplicate conditions of service, it is impossible to get too much information or too much help.

In regard to the questions asked by Mr. Conradson, I would say that I am now planning a different method of supply of lubricant, though my reference in the presentation of the paper was to *rate* of feed rather than to *method* of feed of the oil. I hope that eventually we can investigate both the rate of feed and the method of feed. Mr. Conradson raises a question in regard to the open ends. I think I stated that the test records quoted were made with open ends, but we have had flanges made for the purpose of making it possible to close the ends. When these flanges are put on it will be possible to make tests not only as shown here, but also with any value of pressure in the outer ends of the cylinder, either atmospheric pressure without air, or intermediate pressure without air. In order to work under constant high temperature, which seems to be a very much more severe condition of service than the ones usually found in practice, we are proposing to put on an electrically heated coil over the rubbing surfaces, so that a considerable amount of heat can be supplied inwards, and so that the temperature of the inner wall shall be excessively high and constant, or as nearly constant as it can be made.

In regard to a question as to the difference between oil G and oil T, I would say that the two samples were bought with the idea that they should be as nearly as possible duplicates as to their physical characteristics, as these characteristics are

Mr. Flowers. now measured. The maker of these oils believed that there were some differences in regard to their service characteristics. It was my idea to begin with a pair of materials that were supposed to be duplicates, test them and see whether they were duplicates as regards their friction coefficient and then search for the cause by examining in detail each of the physical characteristics. This part of the test is still in progress.

Mr. Conradson. MR. CONRADSON.—What means are provided for determining the pressure of the piston rings against the cylinder walls and of the piston against the packing rings, or are these pressures not known?

Mr. Flowers. MR. FLOWERS.—The piston ring pressure was measured by the process of closing the ring by pulling the ends together with a spring scale before putting it into the cylinder, and again after it is taken out. This method is open to the objection that there is a slight change in the modulus of elasticity with temperature. It is also open to the objection that it is possible that there may be steam on the wall of the cylinder and also back of the ring. At one time I made some experiments with a small piece of apparatus in which inlets were made by drilling into the piston, and arranging a number of them so that any one of the piston rings could have its back exposed either to the pressure of the atmosphere or to the pressure of the steam on the inside wall. A small difference was observed in the amount of friction at those two extreme conditions. Probably, then, the conditions of service are somewhat intermediate between these two, and there is probably some pressure exerted both on the back and on the face. There may be a little excess on either the back or the face, depending on the amount of leakage. As to leakage, it also occurred to me that this form of construction might be used for measuring the amount of leakage past piston rings. Leakage should be measured with the machine in motion. The leakage is exceedingly small when the machine is at a standstill; it is very much increased when the machine is in motion. The condition of the slipping of the rings along the surface, and variations in the uniformity of the cylindrical wall, make for greater leakage.

ELASTIC LIMIT.

By T. D. Lynch.

SUMMARY.

The term "elastic limit" has come to be used with so many interpretations that serious confusion has resulted.

The following ten distinct terms are in common use: (1) proportional limit, (2) elastic limit, (3) permanent set, (4) true elastic limit, (5) apparent elastic limit, (6) commercial elastic limit, (7) elastic limit by extensometer, (8) elastic limit by drop of beam, (9) elastic limit by the dividers, and (10) yield point.

The first (proportional limit) is represented at point P and the tenth (yield point) at point Y of Fig. 1, the other eight designations being located more or less indefinitely between these points, depending on the interpretation peculiar to different engineers.

The seriousness of the condition is shown in Fig. 2 and Table I, which illustrate variations that may be found in samples from the same bar of steel when subjected to different working and heat-treating conditions. A description is given of the apparatus and methods used in the laboratories of the Westinghouse Electric and Manufacturing Co. A special device for measuring the elongation of a wire 240 in. between gage points is also described, the readings being made to 0.0001 in. in the total length or 0.000000417 in. per inch of gage length.

The following conclusions may be drawn:

1. Commercial accuracy can be obtained with a two-point contact instrument, reading to 0.0001 in. per inch of gage length with the testing machine running continuously up to the yield point.

2. It is practical to use an extensometer in commercial testing as well as in research investigations.

3. This society should establish one or more practical methods of making extensometer tests in commercial laboratories so that the members of the Society and those who look to us for such information can make tests in any laboratory and reach a common conclusion.

This would ultimately result in simplifying, standardizing, and making definite, the results of tests regardless of where and by whom made.

ELASTIC LIMIT.

By T. D. Lynch.

The term "elastic limit" has come to be used with so many interpretations that when we hear it we at once ask ourselves what it means, and what we are to understand when told that the elastic limit shall have a given value. This confusion prevails even with testing engineers and metallurgists as well as with civil, electrical, and mechanical engineers, and other users of materials of construction.

In 1912, Mr. H. F. Moore presented a valuable paper on "The Physical Significance of the Elastic Limit" to the Sixth Congress of the International Association for Testing Materials at New York, in which he gave four distinct meanings of the term "elastic limit" and sounded the keynote in saying that "the indefiniteness of the term 'elastic limit' makes the establishment and use of a distinctive nomenclature highly desirable."

Last year the report of Committee E-1, accepted by the Society, defined the elastic limit to be the "least load per square inch which produces a permanent set as indicated by an extensometer." The committee further stated that this determination was rarely made in the commercial testing of materials.

The definition selected by the committee is correct and well chosen, but while the elastic limit is rarely determined as defined by the committee, this term is in very general use and the tendency is towards further confusion rather than clarification.

A study of a stress-deformation diagram of a tension test, as illustrated in Fig. 1, is introduced in order that we may more easily understand and appreciate the existing conditions.

The elongation is proportional to the load applied up to point P, which point represents the limit of proportionality or "Proportional Limit." Above point P there is a slight increase in the ratio of elongation to the load applied until Y, the yield point, is reached. Here there is a rapid and persistent further increase in the elongation, even though no further load is applied.

Further loading produces a yielding of the test specimen up to M, the point of maximum stress, and this is followed by a reduction in the loading until R, the point of rupture, is reached.

The elastic limit, as defined by Committee E-1, lies above but close to point P, whereas the elastic limit as understood by many users of this term, and I may safely say, by many members of this Society, lies at some indefinite point on the curve, either at P, between P and Y, or at Y.

We are accustomed to hear the free use of the following ten distinct terms, namely, (1) proportional limit, (2) elastic limit, (3) permanent set, (4) true elastic limit, (5) apparent elastic limit, (6) commercial elastic limit, (7) elastic limit by extensometer, (8) elastic limit by drop of beam, (9) elastic limit by the dividers, and (10) yield point.

The first term, proportional limit, is very generally understood to be at point P on the curve, and the tenth term, yield point, at the point Y, whereas the other eight designations are located on the curve, more or less indefinitely, between the points P and Y, depending upon the interpretation peculiar to different engineers.

Fig. 2 is shown to illustrate characteristic stress-deformation curves of tests made on the same rod of steel, the several test specimens differing only in heat treatment. The samples were taken from a rod $\frac{3}{4}$ in. in diameter, having a chemical analysis of carbon, 0.15, manganese, 0.41, phosphorus, 0.006, and sulfur, 0.065 per cent. The rod was cut in 5-in. lengths from which were prepared six test specimens, A, B, C, D, E and F. The treatments given to these specimens were as follows:

Specimens A and B, no treatment;
Specimen C, annealed at 825° C.;
Specimen D, quenched in water from 810° C. and then drawn at 500° C.;
Specimen E, quenched in water from 650° C.;
Specimen F, quenched in water from 860° C.

The physical test results are given in Table I.

The values for yield point were determined by the dividers and those for apparent elastic limit were determined by the

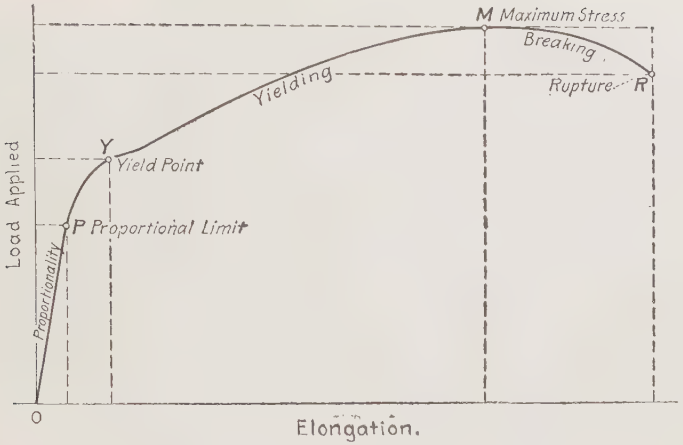

FIG. 1.—Stress-Deformation Diagram of a Tension Test.

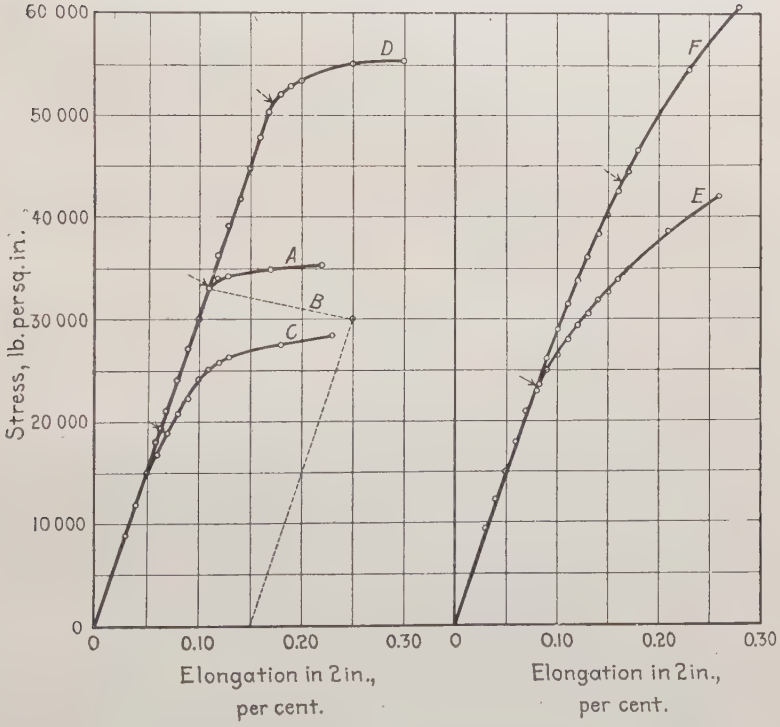

FIG. 2.—Variations in Specimens from the Same Bar Due to Different Heat Treatment.

method described by J. B. Johnson.[1] The test curves were plotted as shown in Fig. 3. The line AB is drawn tangent to the curve at A. The distance BC equals one-half the distance FB. The line AC is drawn through the points A and C. The line DG is drawn parallel to line AC and tangent to the curve at E. The apparent elastic limit is located at the point of tangency, E.

Attention is called to the difference in the ratio of yield point to tensile strength and apparent elastic limit to tensile strength as shown in Table I. These differences are not exaggerated, but are such as are frequently found in tests of steel as well as other metals. Here we have differences between the

TABLE I.—PHYSICAL TEST RESULTS ON SPECIMENS WITH DIFFERENT HEAT TREATMENTS.

Specimen.	Tensile Strength, lb. per sq. in.	Yield Point, lb. per sq. in.	Apparent Elastic Limit, lb. per sq. in.	Elongation in 2 in., per cent.	Reduction of Area, per cent.	Ratio of Yield Point to Tensile Strength, per cent.	Ratio of Apparent Elastic Limit to Tensile Strength, per cent.	Difference of Ratios.
A	52 100	34 500	33 250	45.0	70.3	66.2	63.8	2.4
B	51 625	.....	.....	41.0	68.2	....	....	....
C	50 000	31 000	19 000	38.5	72.9	62.0	38.0	24.0
D	72 250	58 500	51 250	22.2	70.3	81.0	71.0	10.0
E	68 375	49 500	23 500	29.2	65.9	72.4	34.4	38.0
F	123 425	107 750	43 500	4.5	20.6	87.2	35.2	52.0

NOTE.—A quench-bend test was also made on the full-size $\frac{3}{4}$-in. round, the treatment being the same as that of test specimen C, in which the piece was bent flat on itself without showing signs of fracture.

yield-point ratio and the apparent-elastic-limit ratio to the tensile strength of from 2.4 to 52 per cent, and yet all of the test samples were taken from the same bar of steel. The smallest difference in ratio was found on specimen A, which was tested as received from the hot mill, and the greatest difference in ratio was found on specimen F, which was tested after quenching in water from 860° C.

These tests further illustrate the fact that there is a real difference of varying magnitude between points P and Y of Fig. 1, and emphasize the importance of definiteness in the use of these terms which designate that quality of steel or other

[1] "Materials of Construction," p. 18.

material, under stress, which determines the load that may safely be put upon it.

Again referring to Fig. 2, curves *A* and *B* illustrate an important phenomenon occurring at or just above the proportional limit. Tension tests were made on two samples cut from the same rod and without additional treatment. We would expect similar results to be obtained from the two samples.

Specimen A was tested in the usual manner, and the apparent elastic limit was found to be 33,250 lb. per sq. in. Specimen B was tested by first applying a load of 30,000 lb. per sq. in., this load being left on for 30 minutes and then removed, the extensometer returning to zero. The loading was then continued in

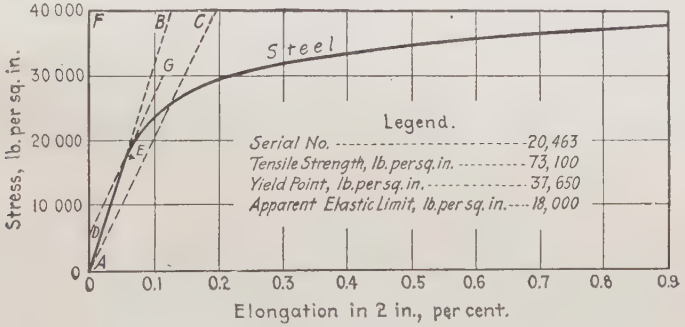

FIG. 3.—Method of Locating Apparent Elastic Limit.

increments of 500 lb. per sq. in. until a stress of 33,000 lb. per sq. in. had been reached, the extensometer in each case returning to zero when the load was removed. Upon the addition of a further increment of 100 lb. per sq. in., the extensometer indicated that the proportional limit had been exceeded. The specimen at once began to stretch, and in less than a minute it had stretched 0.15 per cent. No additional load had been added, but on the contrary, the load had fallen to 30,000 lb. per sq. in., as shown by the dotted line *B*, in Fig. 2.

These tests were made in the physical laboratory of the Westinghouse Electric and Manufacturing Co. on a 150,000-lb. Olsen testing machine, in accordance with the usual practice of the laboratory, as follows:

The standard test specimen adopted by the Society is used,

except that the shoulder adjacent to the threaded ends is cut to $\frac{5}{8}$ in. in diameter, to provide room for clamping the extensometer in position. To simplify the computation of the unit stress, the diameter of the reduced section is made 0.505 in. instead of 0.500 in. This gives an area of 0.2 sq. in.

A modified form of the Ewing extensometer is used, as shown in Fig. 4. The original instrument was modified according to our own designs, to give an open side so that the instrument can be removed from the test specimen without taking it out of the machine. The distance between prick points is 2 in. instead of $1\frac{1}{4}$ in. This instrument is graduated to measure directly to 0.0001 in. and the readings are observed through a microscope.

For the convenience of the operator, a card, graduated in spaces equal to the spaces on the beam, is mounted on the poise of the testing machine and in front of this is mounted a magnetically operated prick point, so arranged that a record of the load on the beam can be made by simply pressing the contact button. This feature is shown in Fig. 5.

The extensometer is attached to the specimen by means of two pairs of sharp thumb screws, so spaced that the length of the specimen included between the points is exactly 2 in.

The poise on the testing machine is set at zero and the cross-hairs of the extensometer are adjusted to read zero. The testing machine is then run at a speed of 0.034 in. per minute. When the extensometer indicates an elongation of 0.0002 in., the operator observing the elongation presses the button and records the load on the card. Likewise at 0.0004, 0.0006, 0.0008 in., etc., until the elastic limit of the material has been exceeded. The machine is then stopped and the instrument removed, thus completing the part of the test requiring the use of the extensometer.

The dividers are next placed in the holes made by the prick points on one side of the test specimen and the machine is run at a speed of 0.06 in. per minute until the stretching increases rapidly as indicated by one end of the dividers leaving the hole. This reading is recorded as the yield point.

The machine is then run at a speed of 0.093 in. per minute until the maximum load has been reached and the test specimen begins to show a reduction in sectional area. The speed is then increased to 0.66 in. per minute until the specimen is ruptured.

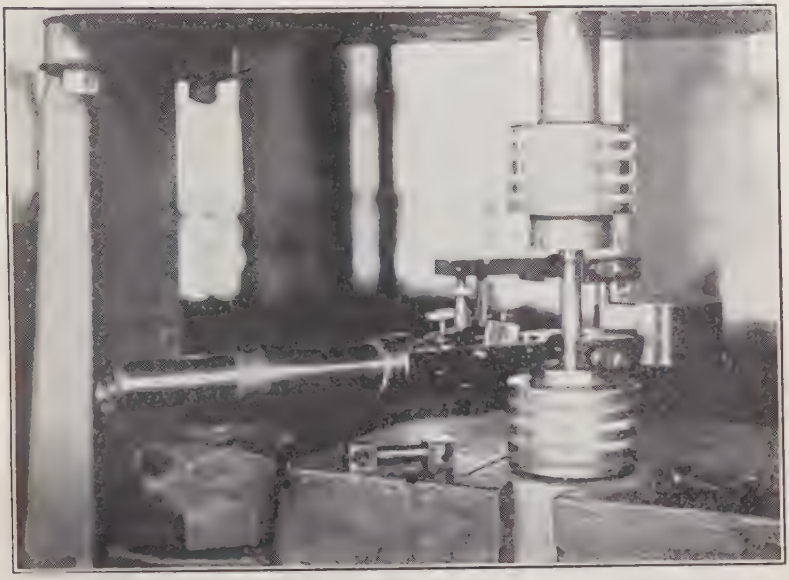

FIG. 4.—Modified Form of Ewing Extensometer.

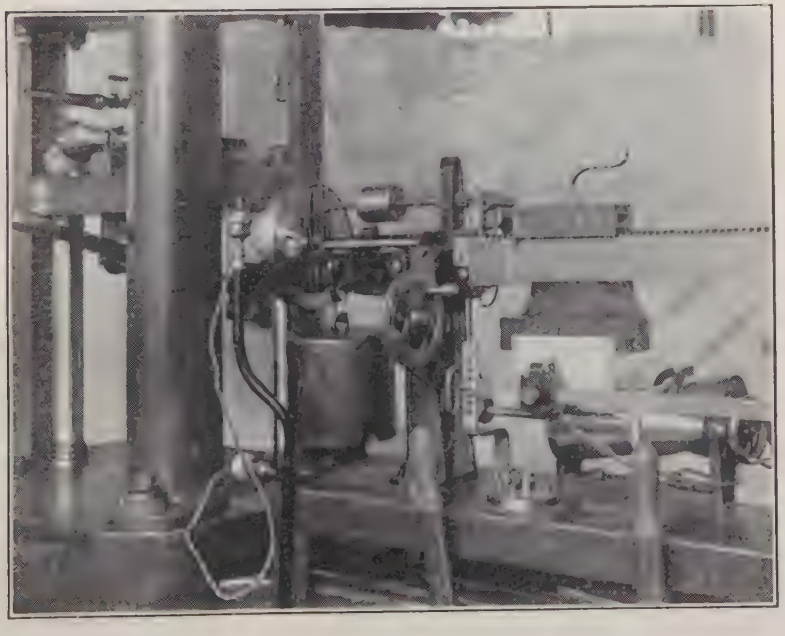

FIG. 5.—Attachment for Recording the Load on the Scale Beam.

The procedure outlined above is followed in the testing of steel, but when testing brass the following slight modification is made in the speed of the machine:

After the proportional limit has been passed, a speed of 0.093 in. per minute is maintained up to the yield point and the

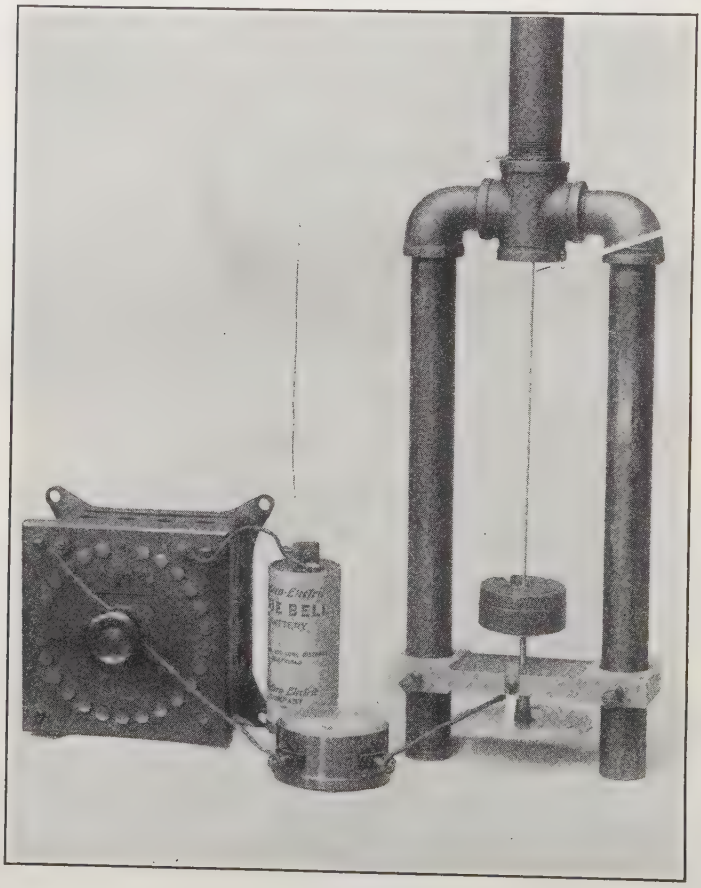

FIG. 6.—Apparatus for Testing 240-in. Samples of Wire.

speed then increased to 0.66 in. per minute until the specimen is ruptured.

The test method described may seem unnecessarily tedious to some, while to others it may seem quite too crude.

We have found that the apparent elastic limit determined in this way is useful for at least two reasons:

1. It is a reliable and sufficiently accurate guide to indicate how much stress can safely be applied to materials subjected to alternating or intermittent stresses;

2. It is a definite value which can be duplicated by different operators.

The apparent elastic limit thus obtained has stood the test

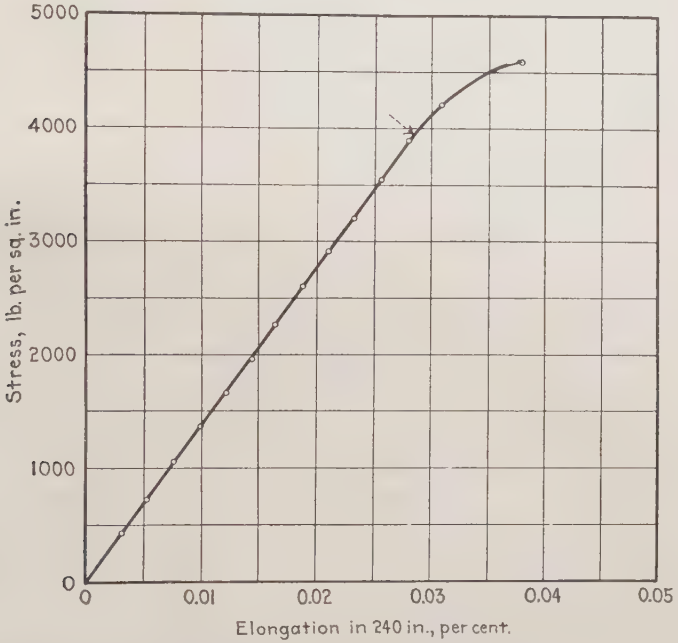

FIG. 7.—Stress-Deformation Diagram of Annealed Copper Wire.

for a number of years and so far has not been found wanting, while on the other hand, the yield-point determination has been found of little value, especially in the application of those materials which have a long sweeping curve between points P and Y, as shown in curves E and F of Fig. 2.

A further series of tests was made on one specimen each of copper wire, iron wire and steel wire, in order to more closely study the shape of a stress-deformation curve when

tested with an accuracy of 0.0001 in. in a gage length of 240 in., or 0.000000417 in. per inch of length.

The apparatus for this test consisted of a 2-in. iron pipe of sufficient length to test a sample of wire 240 in. (20 ft.) long, suspended inside of it. The wire was clamped rigidly at the top of the pipe and a weight, which could be varied, was suspended

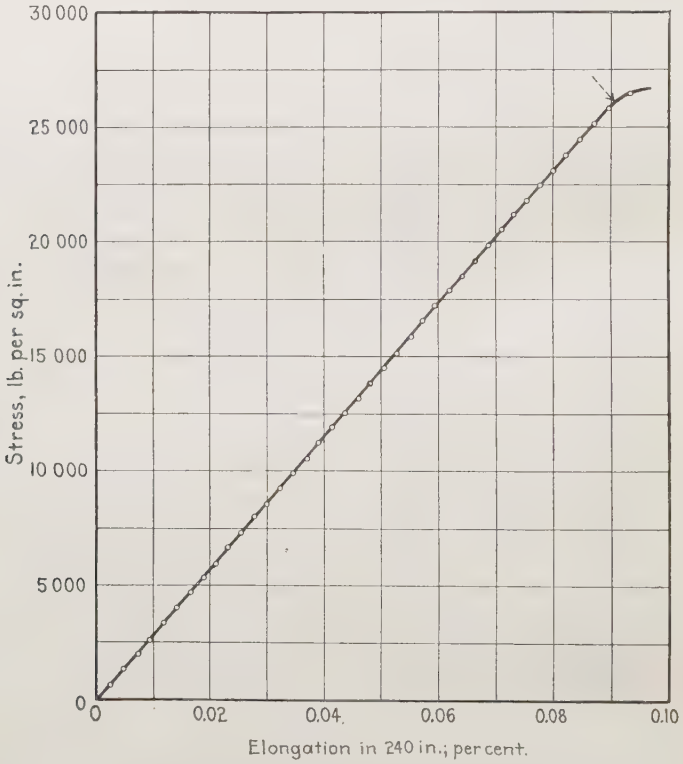

FIG. 8.—Stress-Deformation Diagram of Annealed Iron Wire.

from the lower end of the wire. The lower end of the pipe (Fig. 6) was forked so that a cross-bar could be clamped to it and on this cross-bar was mounted an insulated micrometer screw with a large dial reading directly to 0.0001 in. A steel ball was mounted in the lower side of the weight support, this ball making electrical contact with the end of the micrometer screw.

The temperature was maintained approximately constant by passing a gentle current of air through the pipe from top to bottom. A thermometer was mounted at the lower orifice of the pipe and any change of temperature observed. Provision was made so that weights could be varied in increments of 0.1 lb. for the copper wire and 0.5 lb. for the iron and steel wires.

Wire diameters were selected so that the stresses necessary

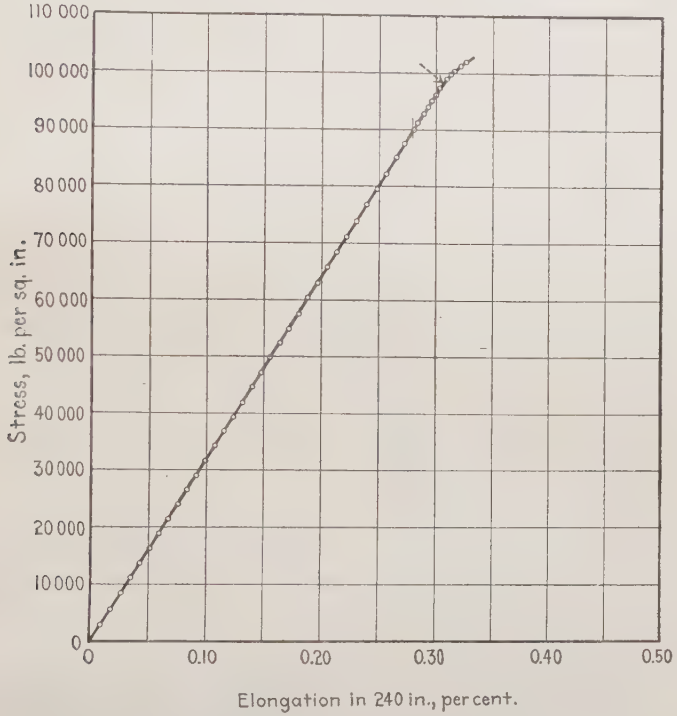

FIG. 9.—Stress-Deformation Diagram of Annealed Steel Wire.

to produce permanent set would be within the capacity of the apparatus. The annealing and straightening of the wire, before test, was accomplished by suspending the wire with a weight attached to it, and then heating the wire electrically until permanently straightened by the suspended weight. The approximate proportional limit and permanent set were determined by applying weights which stressed the wire nearly up to the

predetermined proportional limit and then adding and removing increments of load until a further single increment produced the first permanent set. The greatest load that did not produce a

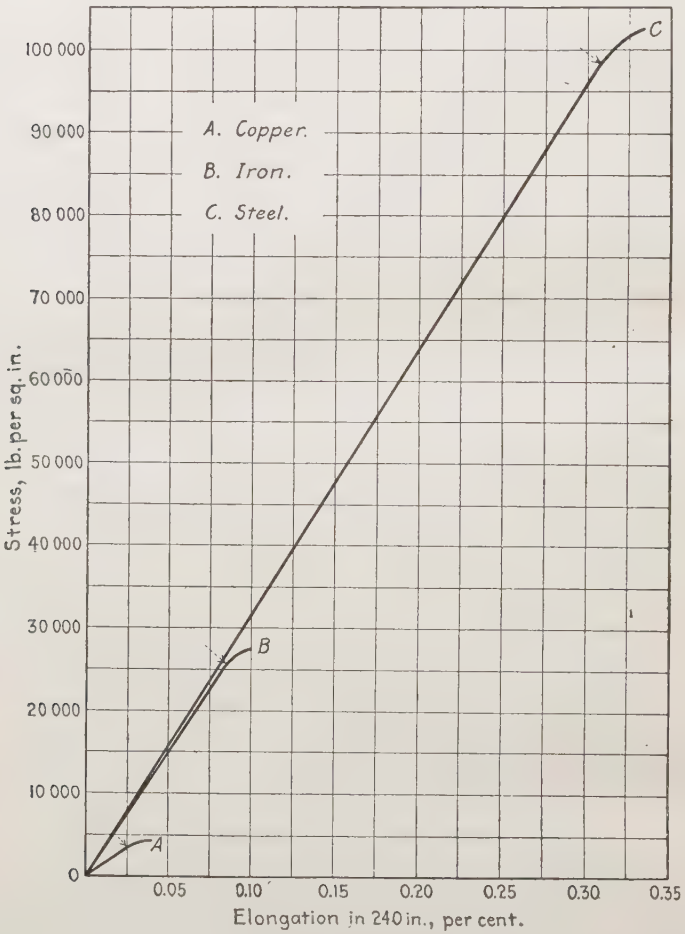

FIG. 10.—Stress-Deformation Diagrams of Annealed Copper, Iron and Steel Wire.

permanent set was truly below the proportional limit, while the least load that did produce a permanent set was equally certain to be above the elastic limit.

The results of these tests are shown separately in Figs. 7, 8 and 9, and all are combined in Fig. 10, in which *A* represents the test on 0.052-in. diameter copper wire annealed, *B* represents the test on 0.034-in. diameter iron wire annealed, and *C* represents the test on 0.029-in. diameter steel music wire annealed. Table II also gives the results of these tests.

Table II shows the relation of the apparent elastic limit to the proportional limit, or a value just below the proportional limit; and the relation of the apparent elastic limit to the elastic limit or a value just above the elastic limit. These comparisons are introduced to show that the values for the apparent elastic limit are definite, cannot be very far from correct, and for all practical purposes are quite safe to use.

The tests represented by Figs. 7, 8, 9, 10 and Table II

TABLE II.—RESULTS OF TESTS ON WIRES.

Sample.	Material.	At or Below the Proportional Limit, lb. per sq. in.	At or Above the Elastic Limit, lb. per sq. in.	Apparent Elastic Limit, lb. per sq. in.
A	Copper.....	3 970	4 280	3 980
B	Iron.....	25 860	26 430	26 080
C	Steel.....	97 640	98 430	98 000

conform very consistently with the routine tests made in the same laboratory with a two-point contact instrument, the machine being operated continuously and with the usual readings of 0.0001 in. per inch of length of test specimen.

Fig. 7 also illustrates the fact that a test can be made sufficiently accurate to show a decidedly straight line in a stress-deformation curve of copper.

There are a number of good machines for testing materials and a variety of extensometers for measuring the elongation of material under test, and it is not the purpose of the writer to advocate standardizing any special make, or even type, of extensometer, but to show that an instrument reading to an accuracy of 0.0001 in. per inch of length between gage points of the test specimens, when properly interpolated, will give good results; and also to say that from experience it has been found

necessary to establish a definite method of making tests and a further definite method of interpolating the results.

We may, conclude therefore, that (1) sufficient accuracy of physical tests of metals can be made with a two-point contact instrument reading to 0.0001 in. per inch of length of specimen under test, to meet the needs of engineers using materials in design; and (2) it is not necessary to stop the testing machine or remove the load until the yield point has been reached.

The foregoing, it is believed, demonstrates the fact that it is practical to use the extensometer in commercial testing as well as in research-laboratory investigations, and we feel, too, that this Society should work out and establish one or more practical methods for making extensometer tests in commercial laboratories, so that the members of the Society, and those who look to us for such information, can make comparative tests in any laboratory.

These would ultimately result in simplifying, standardizing, and making definite the results of tests, regardless of where and by whom made.

DISCUSSION.

Mr. Jesse J. Shuman.—Mr. Lynch has brought out the idea of running the testing machine continuously while extensometer readings are being taken. Our experience in the use of a Berry strain gage during the last year or two has shown the importance of operating the machine without any stops, and we have worked out a plan which gives good results. Mr. Shuman.

Finding the slowest speed of the machine to be too rapid for results that were worth anything, we back-geared the testing machine by belting it from a lathe, and obtained a speed about 40 times as slow as the lowest motor-driven speed. We could then catch the readings accurately without a stop. Later, we arranged to drive the machine by hand. This was done by placing a worm-gear on the driving shaft of the testing machine, which was fitted to a worm on a short shaft operated by a crank. By this simple device we can drive the machine perhaps 100 times slower than by motor, and our practice is to run it continuously while the strain gage is attached, giving intervals of 10 to 15 seconds between points on the dial, each of which represents a 0.0002-in. extension. In this way we can obtain the curve for a two-inch-gage specimen in 10 to 15 minutes, a very good curve, and one that seems to satisfy all the demands of the committee on standard methods of testing. With this experience in mind, I was glad to see the motion passed earlier at this session, referring back to Committee E-1 for reconsideration the two questions of commercial practice in the use of extensometers and of the use of instruments having two-point contact.

To operate the way I have described, conditions must be good. The specimen must be suspended so that no side or twisting strains exist, and the machine must be driven so very slowly that readings can be taken with ease and with a minimum of personal error. Our method requires three people besides the man at the crank: the operator who keeps the weighing-beam balanced and calls out the load at the moment a second person calls the dial reading, and a clerk to record both figures.

Mr. Moore. MR. H. F. MOORE.—I should like to add my experience in the laboratory of applied mechanics of the University of Illinois, to the experience given by the last speaker as to the satisfactory performance of the two-point extensometer in commercial tests, and the possibility of conducting such tests without stopping the testing machine. We have adopted a speed of 0.05 in. per minute of crosshead of machine as a proper speed to use when the machine is not to be stopped. In the discussion which has arisen upon this point, it seems to me that one of the difficulties lies not in the use of the two-point or three-point contact, but in the use of a mechanical device to average the stretch of the test piece on different sides. If such a device is not used, it is necessary to take two or three readings of two or three dials or micrometer screws. Under those conditions, it is not possible to take readings without stopping the testing machine, and it takes 30 or 40 minutes to make a test of a specimen. If a mechanical averaging device is used, such as the extensometer described by Mr. Lynch, and various other extensometers now on the market, such a test can be made without stopping the machine, and it has been our experience that such a method is satisfactory provided the screw head of the machine does not move faster than 0.05 in. per minute.

Mr. Lanza. MR. GAETANO LANZA.—In regard to Mr. Lynch's paper, I will only say that I hope he will state more in detail his views as to conclusion No. 3 of his summary, and also explain just what quantity he desires to determine. Also, as Mr. Olshausen of the Bureau of Standards is present, I should like to have him tell us about his experience.

Mr. Olshausen. MR. G. R. OLSHAUSEN.—I should like to say a few words in regard to the accuracy of determining proportional and elastic limits.

In the three-point method, referred to by Mr. Lanza, on plotting the observation, the stress-deformation lines will not be parallel unless the test piece is properly centered in the machine. The proportional limit obtained by plotting the averages of three corresponding extensions of a not well-centered piece agrees fairly well with that obtained from a well-centered piece of the same material. However, no matter what method may have been used in the determination of the proportional or

elastic limits, these quantities, as any other physical quantities, will always be affected by certain errors which depend on the kind of material, the measuring devices employed, the testing machine and the observer. Mr. Olshausen.

For example, for a material with a sharply defined proportional limit, it may not be possible to determine this limit with the same accuracy if stress increments of 5000 lb. per sq. in. are used as if stress increments of 1000 lb. per sq. in. had been employed. Again, in the case of certain steels for which the rate of deviation of the stress-deformation curve from its straight portion is small, nothing can be gained in the precision of a proportional-limit determination by decreasing the stress increments from 5000 to 1000 lb. per sq. in. The proportional limit is usually determined from a plot on which the observed values have all been assigned the same weight in drawing the stress-deformation curve. That this procedure is not correct for accurate work is clear, since the larger elongations have been determined with a greater degree of precision, with a given measuring device, than the lesser ones. For example, if a load of 10,000 lb. extends a test specimen 0.001 in. and the micrometer readings are correct to 0.0001 in., we have an accuracy of one in ten. If, on the next application of the load, the test specimen is extended 0.002 in., by a load of 20,000 lb., the accuracy of this measurement will be one in twenty. Therefore, the points on the stress-deformation curve lying nearer to the origin should not be given the same weight in determining the slope and position of the straight part of this curve as the more remote ones. As the proportional limit on a plot depends on the location of the straight portion of the stress-deformation line, it is clear that it is not correct to assign the same weight to all observations in determining this quantity. It is not easy to take account of the weights of the observation in a graphical determination of the proportional limit, but methods exist by means of which this can be done, at least for the straight-line part of the stress-deformation curve. They are, however, too complicated for commercial work.

If the observations are taken on a two-inch gage length with a micrometer graduated to thousandths of an inch, and reading by estimation to ten-thousandths of an inch, the read-

Mr. Olshausen. ings taken by two observers usually agree within one or two ten-thousandth of an inch, if the load is constant and the temperature of the micrometer does not vary more than five degrees.

All of these questions must be taken into account in an accurate determination of such quantities as modulus of elasticity, elastic and proportional limits.

Mr. Moore. MR. MOORE.—If I may add another word, I wish to state that in referring to the practice of the laboratory of applied mechanics of the University of Illinois, I was not referring to technical practice, but to the practice for commercial tests. It would be a very difficult matter to make definite specifications for technical testing. At Illinois, within a few months, we have made a test of a specimen on which we took readings, not on three gage lines, but on twenty-six gage lines, and we found that a cross-section of the test piece which was a plane before loading did not, in general, remain a plane after loading, and that the whole matter of precise stress-distribution was very much more complicated than could be taken care of even by a three-point, three-gage-line instrument.

The discussion brings out clearly the fact that we must distinguish between methods practicable for commercial testing and methods necessary in certain refined technical tests, and it is the speaker's belief that there are reliable methods of making extensometer tests which are practicable in commercial work.

Mr. Shuman. MR. SHUMAN.—I wish to add a word for fear my former remarks might be misconstrued. When I attempt to defend a two-point contact instrument, I am speaking exclusively of the use of a standard turned test piece, suspended in ball-and-socket joints so that there can be no side strains. It is clearly out of the question to obtain any of these values on a test piece that is merely held in the grips of the machine, and if Mr. Lanza's objection to the use of the two-point extensometer is based upon a test piece that is gripped in the machine, I have no criticism to offer. I am simply defending the use of a two-point instrument when applied to a test piece that has no transverse strains in it.

MR. OLSHAUSEN.—All of the tests that we made for Mr. Lanza at the Bureau of Standards were made with the ball-and-socket joint, but when the test piece is only slightly out of line, the angle of repose is such that it does not allow that test piece to line up exactly. To obtain good alignment, we apply a small load and tap the test piece, until there is no further reduction of load; that is, we get the shortest distance between the points of application of the load. Another way is to draw the curves and see whether all three curves have nearly the same slope. If they are not parallel, release the specimen and try again. We found that this method of tapping until we get the least distance between the two points of attachment, gave us the quickest results. Mr. Olshausen.

MR. MONT SCHUYLER.—Two years ago I presented a paper here on "Spherical Bearings."[1] At that time I called attention to the necessity of a three-point extensometer reading. It is necessary, even if you use ball-and-socket joints, and standard test specimens. If you will examine the elastic characteristics of any testing machine, even those with guide columns, you will find that there is enough wobbling of the head (not only in the first period of the test, but throughout the test) to make a three-point reading necessary. Mr. Schuyler.

MR. R. S. MACPHERRAN.—Mr. Lynch says in his paper, that "the smallest difference in ratio was found on specimen A, which was tested as received from the hot mill, and the greatest difference in ratio was found on specimen F, which was tested after quenching in water from 860° C." Is that always the case and are these differences constant? Is it always found that the smallest difference in ratio occurs in the normal material without treatment, and the greatest difference in the quenched material? Mr. MacPherran.

MR. OLSHAUSEN.—That depends entirely on the grade of the material. Mr. Olshausen.

May I have another word? I think with two-point contact, the apparatus has a chance to adjust itself just about as well as with three-point contact. Three mechanically distinct readings at three points will give more accuracy, I think, than readings at two points.

[1] *Proceedings*, Am. Soc. Test. Mats., Vol. XIII, p. 1004 (1913).

Mr. Lynch. MR. T. D. LYNCH (*Author's closure by letter*).—In answer to Mr. Lanza's request for more detail with reference to conclusion No. 3 of my summary, I would say that at the present time there is no standard method or methods of making extensometer tests and we are in great need of one that is practical to use in commercial testing as well as in Research Laboratory investigations. We need to know the working value of materials in service; and in order to test with this object in view, we should be able to simplify, standardize and make definite the practical results of test regardless of where and by whom made.

In answer to Mr. MacPherran's question, I wish to say in reply that tests A to F, inclusive, were introduced to illustrate characteristic stress-deformation curves that we encounter in testing and to show that remarkably different values may be found in the same piece of steel when treated differently. The relative values shown in these tests are not necessarily constant under different conditions, since the normal condition of steel as received from the mill will vary with the practice of the mill. The temperature at the time of the finishing pass through the rolls, as well as the rate of cooling on the hot bed, tends to vary the test results. Properly heat-treated samples, however, should give the form shown in curve *D*, Fig. 2.

THE FAILURE OF MATERIALS UNDER REPEATED STRESS.

By H. F. Moore and F. B. Seely.

SUMMARY.

Nearly all our ideas of the action of materials under stress are derived from considerations of static action. The action of materials under repeated stress differs from static action in important respects.

Under stresses slightly lower than the elastic limit as ordinarily determined, materials fail under oft-repeated stress, and the relation of stress at failure to number of repetitions may be expressed by an exponential formula, as shown by Basquin and others.

A proposed explanation of this formula is given, based on the structural damage done to material by inelastic action during application and removal of load.

For low stresses no positive evidence of a fixed endurance limit has been found for commercial materials, and various methods used for locating such a supposed limit give varying results from the same test data.

A modification of the exponential formula is proposed for low stresses.

Tentative values of constants for the proposed formulas are given and also some general discussion on action under repeated stress.

THE FAILURE OF MATERIALS UNDER REPEATED STRESS.

By H. F. Moore and F. B. Seely.

Behavior of Materials.

Materials under Static Stress and under Repeated Stress.—The behavior of materials under repeated stress shows important variations from the action under static stress. Nearly all the ideas of repeated stress have been developed from considerations of static loading. One very common idea is that for any given material there is a definite elastic limit below which the behavior of the material is perfectly elastic. Under static loads such a conception may be regarded as exact without involving serious error, though careful writers on the mechanics of materials have for a long time recognized that no absolute elastic limit has ever been fixed for any material.[1] In structures under static load local stresses of considerable magnitude—frequently beyond the yield point of the material—exist without producing any appreciable effect on the stability or the deformation of the structure as a whole, and such stresses are frequently neglected in structures subjected to static load. If, however, the load on a structural part or a machine member is repeated many times such local overstress may cause a crack to start which, spreading, eventually destroys the member; or inelastic action too small to be detected even by delicate static tests of material may by cumulative action cause serious damage under oft-repeated loading.

An illustration of the difference between static and repeated loading is furnished by the action of wire ropes bent around sheave wheels. The fiber stress due to bending is high and frequently causes inelastic action, which, however, is confined to a small portion of each wire. This inelastic action is very difficult to detect by means of static tests, but as the wire is

[1] Thurston, "Text-Book of Materials of Construction," p. 348; Burr, "The Elasticity and Resistance of Materials of Engineering," p. 200; Wawrziniok, "Handbuch des Materialsprüfungwesen," p. 9; Unwin, "The Testing of Materials of Construction," p. 13.

repeatedly bent around sheave wheels this high local stress starts cracks which eventually cause rupture of individual wires. In this case, as in many others, the conception of perfect elastic action, allowable for static loading, must be discarded for repeated loads.

Materials under Repeated High Stress.—For a range of fiber stress extending from the yield point of the material (for brittle materials the ultimate strength) down to a stress slightly lower than the elastic limit, as determined by laboratory tests of the usual precision, repeated stress will cause failure, and there seems to exist a fairly definite relation between fiber stress and the number of repetitions necessary to cause failure. This relation was pointed out by Basquin[1] before this Society. It may be expressed by the formula:

$$S = KN^q$$

in which S = intensity of fiber stress in pounds per square inch, N = the number of repetitions of stress to cause failure, and K and q are experimentally determined constants. A similar relation was noted later by Eden, Rose and Cunningham[2] and by Upton and Lewis.[3] Whether for still lower stresses such a law holds, or whether there is an "endurance limit" below which failure will not occur under any number of repetitions of stress, however many, is a question which will be discussed later.

Within the stress limits named above, if material is subjected to a cycle of stress involving application and removal of load, delicate measurements of deformation will show that the relation between stress and deformation is represented not by a single straight line, but by two curved lines, one for application and one for removal of load. Even if such deviation cannot be detected after a single cycle of stress it has been shown by Bairstow[4] that the deviation may become appreciable after several thousand repetitions, and that the stress-deformation curve for a cycle of stress after a few repetitions becomes a closed

[1] "The Exponential Law of Endurance Tests," *Proceedings*, Am. Soc. Test. Mats., Vol. X, p. 625 (1910).

[2] "The Endurance of Metals," *Proceedings*, Inst. Mech. Engrs. (British), Parts 3 and 4, p. 389 (1911).

[3] "The Fatigue Failure of Metals," *American Machinist*, Oct. 17 and 24, 1912.

[4] "The Elastic Limits of Iron and Steel under Cyclical Variations of Stress," *Philosophical Transactions*, Royal Soc., A Vol. 210, p. 35 (1910).

curve with a general form like that shown in Fig. 1 (*a*). These curves of stress-cycles resemble the "hysteresis" curves of magnetic material; the area enclosed by the loop represents energy lost during a cycle of stress, and this loss of energy is spoken of as "mechanical hysteresis."

If mechanical energy is dissipated during a cycle of stress it would seem that the lost energy must be transformed into heat, that there must be some form of internal friction in the material, that wear and structural damage take place, and that

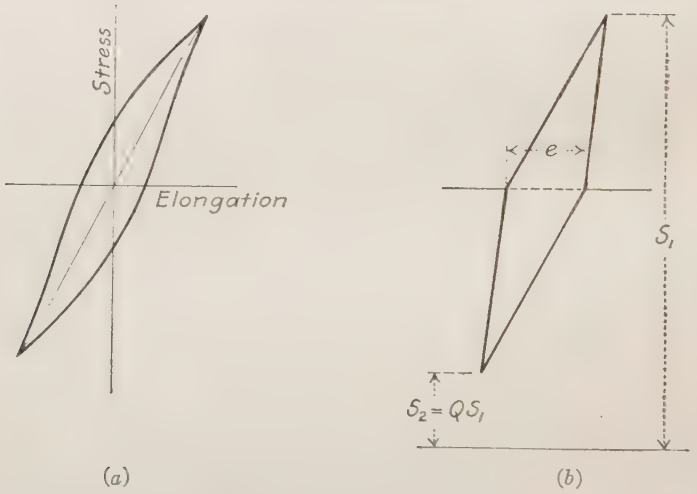

FIG. 1.—(*a*) General Form of Stress-Deformation Curve for a Cycle of Stress. (*b*) Approximate Form of Curve.

if the action is continued long enough the material will be ruptured. It would seem reasonable to consider the amount of structural damage during a cycle of stress to be proportional to the energy transformed into heat, or, in other words, to the area of the mechanical hysteresis loop. The shape of this loop remains similar to the shape developed under early cycles of stress. Referring to Figs. 1 (*a*) and (*b*), this area is seen to be very nearly equal to that of two triangles placed base to base, or to $\frac{1}{2}(S_1 - S_2)e$. If S_2 is denoted by QS_1 (Q is negative if the cycle involves reversal of stress) the structural damage done during an early cycle of stress ($\triangle'$) is given by the equation:

$$\triangle' = AS_1(1-Q)e$$

in which A = some constant and e = the width of the hysteresis loop. An examination of the various tests which have come under the writers' notice, especially those of Bairstow,[1] indicate that e is some function of the range of stress $(1-Q)S_1$ and may be taken as proportional to $S_1^{p}(1-Q)^{p}$ in which p is some constant. The structural damage done during a cycle of stress is then given by the equation:

$$\triangle' = A' S_1^{(1+p)}(1-Q) \qquad \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

in which A' is some constant to be determined experimentally.

The damage done by successive cycles is not the same. An examination of Bairstow's results, of the results of tests by Sondericker,[2] and of tests in the Laboratory of Applied Mechanics

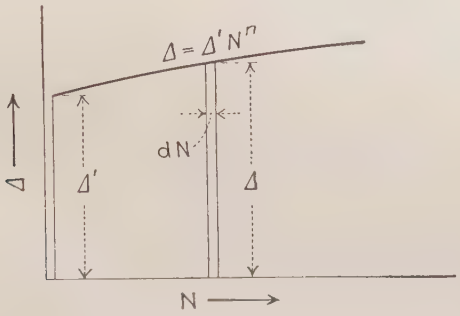

FIG. 2.—Relation between Damage per Cycle and Number of Repetitions of Stress.

of the University of Illinois indicates that the damage $\triangle$ changes slightly with successive cycles and that the relation between damage per cycle, $\triangle$, and the number of repetitions of stress, N, may be represented by the equation:

$$\triangle = \triangle' N^{n}$$

in which $\triangle'$ = the damage done per cycle during the earlier cycles of stress, and n = a constant which has a very small numerical value. This equation is represented by a curve similar to that shown in Fig. 2.

[1] "The Elastic Limits of Iron and Steels under Cyclical Variations of Stress," *Philosophical Transactions*, Royal Soc., A, Vol. 210, p. 35 (1910).

[2] "Repeated Stresses," with a discussion by J. E. Howard, *Technology Quarterly*, March, 1899.

The total structural damage done by any number of repetitions, N, may be denoted by T, and will be represented by the area under the curve up to the value of N or by the expression $\int_{\cdot}^{\cdot} \triangle \, dN$ which is equal to $\int_{a}^{N} \triangle' N^n dN$ (a is some small number—the first few cycles are apt to be irregular). Integrating the above expression there is obtained:

$$T = \frac{\triangle'}{1+n}(N^{(1+n)} - a^{(1+n)})$$

and as N is large as compared with a this may, without serious error, be written:

$$T = \frac{\triangle'}{1+n} N^{(1+n)}$$

but from equation (1)

$$\triangle' = A' S_1^{(1-p)}(1-Q)^{(1+p)}$$

hence

$$T = \frac{A' S_1^{(1+p)}(1-Q)^{(1+p)} N^{(1+n)}}{1+n}$$

If the structural damage spreads regularly across the most-stressed cross-section of a member, and if the amount of structural damage necessary to cause failure be denoted by W, then at failure under repeated stress:

$$W = T = \frac{A' S_1^{(1+p)}(1-Q)^{(1+p)} N^{(1+n)}}{1+n}$$

Grouping the experimental constants W, A', $1+p$, and $1+n$, denoting $W(1+n)$ A' by $B^{(1+p)}$, and $1+n$ $1+p$ by q, solving for S_1 and dropping the subscript, there is obtained:

$$S = \frac{B}{(1-Q)N^q} \qquad \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

For any given set of experiments with a constant value of Q, the stress to cause failure under repeated stress might be expected to be proportional to some negative fractional power of the number of repetitions, which result is in harmony with the exponential law proposed by Basquin, as a result of his direct study of results of tests under repeated stress.

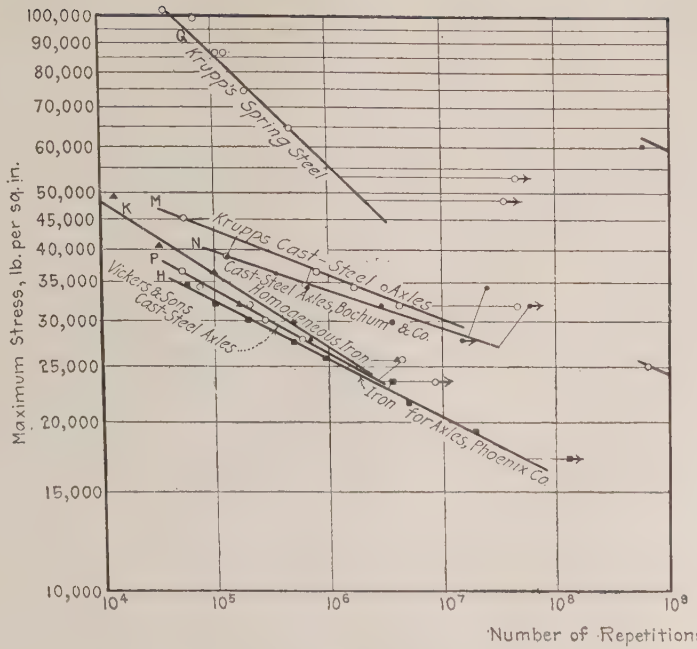

FIG. 3.—Wöhler's Tests.

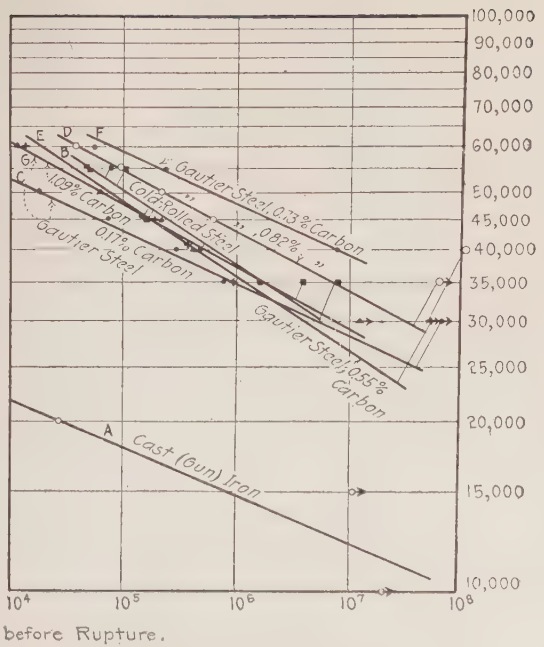

FIG. 4.—Watertown Tests.

Equation (2) may be written:

$$\log S = \log B - \log (1-Q) - q \log N \quad \ldots\ldots\ldots\ldots\ldots\ldots (2a)$$

Equation (2) is represented by a straight line if plotted on logarithmic cross-section paper. As shown in Basquin's paper, also by Eden, Rose and Cunningham, and by Upton and Lewis, the results of nearly all repeated stress tests, if plotted on logarithmic paper, do follow a straight line up to a value of N slightly greater than 1,000,000. In Figs. 3, 4 and 5 are given some test results compiled by the writers, including some not given in Basquin's paper. These results also follow a straight line up to a value of N of about 1,000,000; the corresponding value of S is slightly below the elastic limit which would be determined by a static test of the usual laboratory accuracy. The writers wish to acknowledge the loan of collected test data by Professor Basquin, which was of material assistance.

The analytical discussion of the cumulative damage done by repeated stress, which has been given, seems to yield results in accordance with the results of tests, and is submitted as an explanation of the failure of materials under repeated stress within the stress limits named; that is, for stresses ranging from the yield point (or ultimate strength for brittle materials) down to a stress slightly below the "elastic limit" as usually determined in static tests.

Materials under Repeated Low Stress.—As builders of machines have to design parts to withstand many times one million repetitions of stress, a problem of greatest importance is the determination of the action of repeated stresses lower than those considered in the foregoing paragraph. Is cumulative damage done under these lower stresses, and will they finally cause failure? Or is there an "endurance limit" below which no damage is done to the material, and below which the material will withstand an infinite number of repetitions?

The latter view is the one which has been widely held, and the endurance limit has been regarded as coincident with the "true" elastic limit of a material. In favor of this view may be cited the fact that the number of repetitions of stress necessary to cause failure increases very rapidly as the fiber stress is lowered; that a number of tests have been made in which test

specimens withstood tens of millions of repetitions of stress without failure; and that at low stresses, even with delicate measuring apparatus, there can be detected no signs of structural damage. Various methods have been used for determining the value of the endurance limit for a material; these different methods yield widely varying results, as will be shown in detail later.

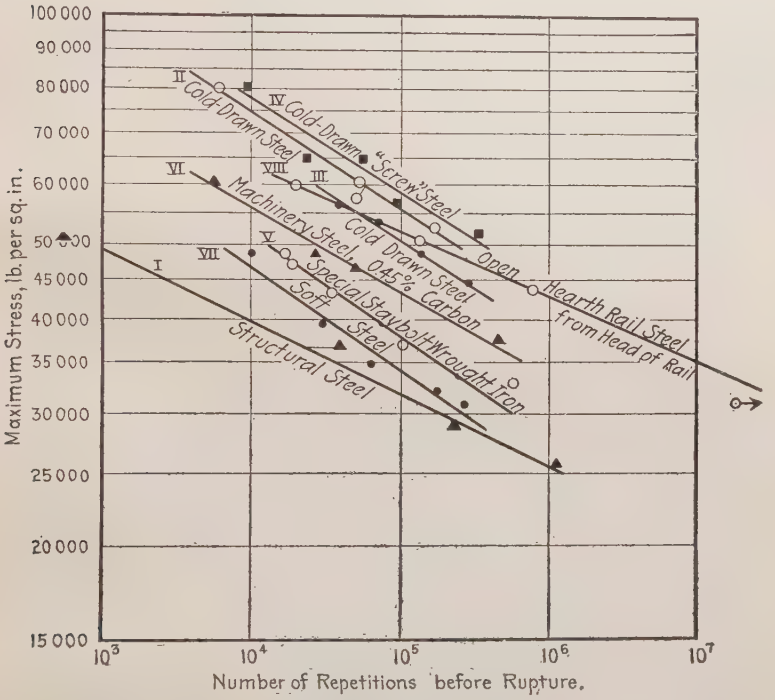

FIG. 5.—University of Illinois Tests.

In favor of the view that damage is done to materials under low stress and that there is a probability of their eventual failure under repeated low stress, the following considerations may be cited:

1. The occurrence of "slip lines"[1] in metal under repeated

[1] Ewing and Rosenhain, "Crystallin Structure of Metals," *Philosophical Transactions*, Royal Soc., Vol. 193, p. 353 (1899); Ewing and Humfrey, "Effect of Strain on the Crystallin Structure of Lead," *Philosophical Transactions*, Royal Soc., Vol. 200, p. 241 (1902).

stress seems to be the result of cumulative damage within a crystal of metal. No sharply defined lower limit has been found, either for the appearance of these slip lines or for their tendency to spread and develop into cracks.

2. The gradual development of permanent set, under repeated stress so low that preliminary static tests had shown no measurable set, seems to the writers to be an indication of damage at low stresses. This development is shown especially by the tests of Bairstow[1] for cycles of stress not involving complete reversal. Bairstow found that the set for any stress gradually increased, though for a single cycle of stress no mechanical hysteresis could be detected; and that after several thousand repetitions the set did not further increase during the test. Whether this set would have shown further increase with an increase in the number of repetitions, or whether increase would have been shown by more delicate instruments, is an undecided question. In the opinion of the writers the significant fact is the cumulative development of permanent set under repeated low stress.

3. The sudden sharp breakages which occur in repeated stress tests, even of ductile materials, would seem to indicate that structural damage may be done to material without any undue deformation of the member as a whole. The fact that no undue deformation can be detected is no sure sign that a material is free from danger of failure under repeated stress.

4. Data of tests involving more than a million repetitions of stress are very few, yet frequently machine parts must be designed to endure several hundred millions of repetitions. The repeated stress problems of the time of Wöhler and Bauschinger were mainly problems of railroad bridge members and other structures and machines which would be called on to withstand only a few million repetitions of stress. From the viewpoint of these earlier investigators experiments under a few million repetitions covered the ground; for machines of to-day reliance on the results of such experiments involves enormous extrapolation of test results. The data seem hardly sufficient for establishing an endurance limit for infinite repetition, or

[1] "The Elastic Limits of Iron and Steel under Cyclical Variations of Stress," *Philosophical Transactions*, Royal Soc., A Vol. 210, p. 35 (1910).

even for repetitions numbering hundreds of millions. Moreover, the results of some tests, if taken alone, seem to indicate that some exponential law of endurance holds up to the limit of experimentation. These unusual tests are discussed later.

5. As instruments of increased delicacy are used in measuring deformation, evidences of mechanical hysteresis are found at lower and lower stresses in static tests.[1] In actual material these evidences have been found at stresses not much above ordinary working stresses. When the cumulative action of repeated stress is considered, the indefiniteness of the elastic limit becomes apparent. While the statically determined elastic limit has some significance for static loading, it apparently has no significance as a criterion of endurance strength.

6. If elastic vibrations are set up in metal test specimens such vibrations soon die out.[2] This dying out would seem to indicate loss of energy in heat, with accompanying internal friction, wear, and structural damage.

The following quotation from the careful work of Bairstow[3] is of interest:

"For equal (reversed) stresses after the specimen had been fixed in position and before it had been loaded in either direction, a reading was taken of the unstrained length. A similar reading was recorded after the tensile loads had been applied and removed, and a third reading after putting on and removing the compressive load. The three readings were alike and indicated complete elasticity within the accuracy of the measurement. The stress, ± 31,000 lb. per sq. in., was then repeated automatically, and for some time the straight line continued to represent the cycle of extensions. As the number of repetitions became greater the 'cyclical permanent set' became measurable and gradually increased until after 19,000 reversals of stress it had become 11 per cent of the original elastic extension. . . . Raising the stress to 33,000 lb. per sq. in. produced an immediate increase in the 'cyclical permanent set' . . . Finally stresses of ± 44,440 lb. per sq. in. were imposed and at 29,280 reversals . . . the width of the hysteresis loop was very great, but even for this case the lines (showing release of loads, both tension and compression) are parallel to the original elastic line. . . .

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[1] Moore, "The Physical Significance of the Elastic Limit," *Proceedings*, Internat. Assn. Test. Mats., Article XXVIII (1912).

[2] Boudouard, "Break-Down Tests of Materials," *Proceedings*, Internat. Assn. Test. Mats., Article V3 (1912); Kelvin, "On the Elasticity and Viscosity of Metals," *Proceedings*, Royal Soc., May 18, 1865; also "Collected Mathematical and Physical Papers," Vol. III.

[3] "The Elastic Limits of Iron and Steel under Cyclical Variations of Stress," *Philosophical Transactions*, Royal Soc., A Vol. 210, p. 35 (1910).

"The behavior of this specimen illustrates the necessity for Bauschinger's hypothesis relating to primitive elastic limits, as the extensometer was incapable of showing the first deviation from elasticity. At a slightly lower range, probably = 28,600 lb. per sq. in., the specimen would have been really elastic, as no number of reversals would have produced a hysteresis loop."

The writers feel that the conclusion given in the last paragraph is more sweeping than is justified by the data, and that the conclusion in such a case (which is typical) should rather have been, that a slightly lower stress applied to the specimen would have required *many more repetitions* in order to make it evident that damage was being done. After a study of the data of many series of repeated stress tests, the writers feel that no "endurance limit" has been surely found below which any material may be relied on for indefinite endurance. It seems to the writers that more repetitions of stress would probably have broken the test specimens, more delicate instruments would probably have shown evidences of inelastic action, rise of temperature, or other sign of eventual failure.

To the writers it seems that a negative argument against the use of a definite endurance limit is furnished by the indefiniteness of its determination. A common method of locating this limit is to plot from test a curve with stresses as ordinates and number of repetitions to cause failure (N) as abscissas. This method is shown in Fig. 6 (a) for a typical set of test results from Wöhler. This curve becomes nearly horizontal at a few millions of repetitions and the horizontal line to which the curve is asymptotic is judged by the eye. The ordinate of this horizontal line is taken as the endurance limit, and in this case gave a value of 18,000 lb. per sq. in.

Another method is to plot stresses as ordinates and values of $1/N$ as abscissas. The endurance limit is taken as the ordinate of the intersection of this curve (extended) with the zero axis. This method is shown in Fig. 6 (b) and for the same test data as the first method gives a value for the endurance limit of 17,500 lb. per sq. in.

A third method is to plot stresses as ordinates and some root of $1/N$ as abscissas. The fourth root of $1/N$ has been suggested by C. E. Stromeyer.[1] Fig. 6 (c) shows the application

[1] "Memorandum of the Chief Engineer of the Manchester Steam Users Association" for 1913.

of this method to the given test data, and the endurance limit is found to be 15,000 lb. per sq. in. Fig. 6 (*d*) has been plotted with values of the eighth root of $1/N$ as abscissas, and the value of the endurance limit thus found is 7,000 lb. per sq. in. This

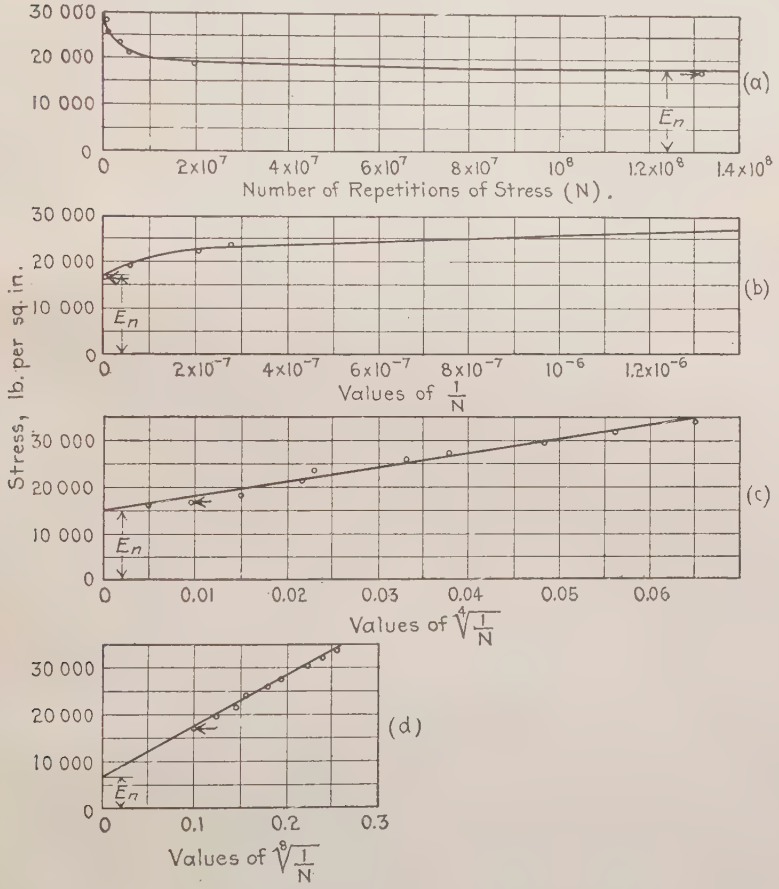

FIG. 6.—Diagrams Showing Variation in Endurance Limit (E_n) with Method of Determination.

series of tests involved one test at 19 million repetitions and one at 132 millions. If these various methods were applied to tests covering no more than one million repetitions of stress the results would show still greater variation.

FORMULAS.

Modification of the Exponential Equation for Repeated Stress.—The exponential equations for repeated stress, (2) and (2*a*), give for high values of N, stresses which are lower than those which material has withstood in several tests. This was shown by Basquin, by Upton and Lewis, and is illustrated in Figs. 3, 4 and 5. Some modification of these formulas would seem to be necessary for low stresses and high values of N. If a test specimen or machine member fails under a few repetitions of high stress the damage done by each cycle of stress is considerable; an appreciable proportion of the cross-sectional area is affected by this damage, and the spread of damage is regular and rapid. If a test specimen or machine member is subjected to cycles of low stress the damage done during each cycle is much less; a much smaller proportion of the area is affected, and the location of the little areas affected is dependent on the homogeneity of the material and the regularity of distribution of stress. For example, a short cylinder under compression has a more uniform distribution of stress under a load sufficiently heavy to cause firm bearing between the cylinder and its bed, than it has under a load so light that the extremely small areas projecting from the end of the cylinder are not all mashed flat. Under low stress the rate of spread of damage becomes somewhat a matter of probability and chance. The affected areas *may* be so grouped that damage proceeds regularly, but probably they will be somewhat scattered; the damage will proceed with corresponding slowness and the endurance of the member will be increased. The writers suggest that the exponential equations, (2) and (2*a*), modified by the addition of a "probability factor," be used for estimating the fiber stress which will cause failure under any given number of repetitions of stress, N, and range of stress, Q. They suggest as such a factor $1+kN^m$ in which k and m are constants to be derived from experiment. The modified exponential formula then becomes

$$S=\frac{B}{(1-Q)N^q}(1+kN^m) \quad \ldots\ldots\ldots\ldots\ldots\ldots (3)$$

$$\text{or } \log S=\log B-\log (1-Q)-q \log N+\log (1+kN^m) \quad \ldots\ldots (3a)$$

These formulas seem rather formidable, but tabular values for $1+kN^m$, and graphical charts for the solution of the formulas are given later which facilitate their use.

The variability in the action of metals under repeated stress is well illustrated by the results of a series of tests of cold-rolled steel made at the Watertown Arsenal.[1] The results of this series of tests is plotted logarithmically in Fig. 7. It will be seen that for the higher values of stress the range of values of

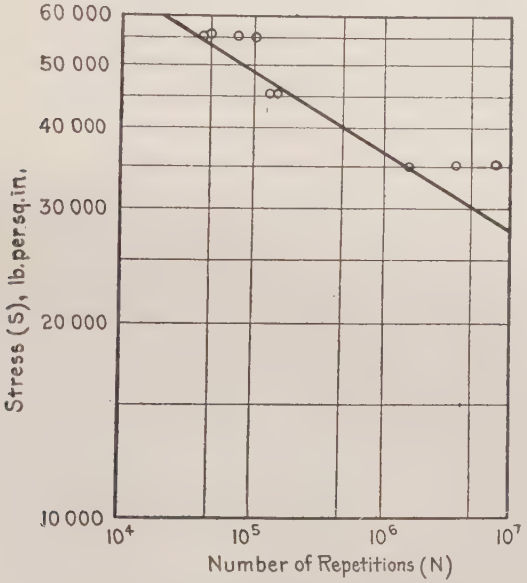

FIG. 7.—Tests of Cold-Rolled Steel. Watertown Arsenal.

N is less than for low values of stress. A straight line fairly well represents the results for minimum values of N; this straight line corresponds to an exponential law, equation (2), and for such minimum values it would seem that the damage caused by repeated stress proceeded regularly. For the other test specimens the damaged areas were more scattered, or for some other reason damage proceeded more slowly, and this effect became more marked at lower stresses.

[1] "Tests of Metals," 1893, pp. 161–165, 170, 171, 176–178.

The objection may be raised to the formula proposed by the writers in that it involves failure of any material under any stress, however small, if that stress is repeated a sufficiently great number of times; and it may be stated that there is no proof that such failure will occur. It has not been conclusively proved that very low repeated stresses will ultimately break materials, but on the other hand, no endurance limit for indefinitely repeated stress has yet been positively established for any material. There is practically no direct experimental evidence concerning the behavior of material under more than 100 million repetitions of stress; there is very little experimental evidence for more than 10 million repetitions, yet some machine parts must be designed to withstand 10 billion repetitions of stress.

The writers believe that a formula which assumes that the same destructive action which occurs under high stresses will continue to act with diminished intensity under low stresses has some indirect evidence in its favor, and is a safer guide for the designer than is a fixed endurance limit below which destructive action is assumed to cease. It should be noted that even for values of N as great as 10 billion, the modified exponential formulas proposed by the writers give stresses which, though somewhat lower than the values usually given for the endurance limit, are yet of very considerable magnitude. This will be discussed under the head of "Constants for the Formulas."

In addition to the above considerations, it should be noted that for very long endurance of machines or structures deterioration due to other causes than repeated stress—wear, corrosion, change of current practice, and the like—becomes a criterion of endurance.

Constants for the Formulas.—The writers have studied the available data of repeated stress tests and submit tentative values for the constants in the formulas proposed, namely, equations (2), (2*a*), (3) and (3*a*).

B has been determined for a number of materials from the data of repeated stress tests. It is the value of the ordinate for $N=1$ (line extended backward)[1] and $Q=-1$ (stress com-

[1] This does not mean that the formula is to be used for values of N as low as 1; the formula is *not* to be used if it gives stresses above the safe *static* working stress.

pletely reversed). In Table I are given tentative values of B for some common metals. The value of Q (ratio of minimum stress to maximum stress) is generally known for any structural part or machine member. If the stress is wholly or partially reversed Q is negative. If Q approaches $+1$ in value, care should be taken that the safe *static* stress is not exceeded; the safe static stress is always effective as a criterion of safety.

The effect of range of stress has been studied in connection with the work of Bauschinger and Wöhler. Equations (2) or (3) gives values of S for complete reversal ($Q=-1$) one-half as great as the values for repetition of stress from zero to a maximum ($Q=0$). This seems to agree fairly well with test results,

TABLE I.—VALUES OF THE CONSTANT B IN THE EXPONENTIAL FORMULAS FOR THE ENDURANCE OF MATERIALS UNDER REPEATED STRESS.

Material.	B	Log B
Structural Steel and Soft Machinery Steel	110 000	5.0414
Cold-rolled Steel Shafting	275 000	5.4393
Steel, 0.45 per cent Carbon	175 000	5.2430
Wrought Iron	100 000	5.0000
Hard Steel	250 000	5.3979
Hard-Steel Wire	400 000	5.6021

but the effect of range of stress furnishes a most promising field for further experimental study.

A value of $q=\frac{1}{8}$ seems to fit fairly well the results of a wide range of tests of various-size test specimens of different metals tested on a variety of machines. Considerable variation from this value may be found, but the writers have been able to find no systematic variation; and as was pointed out by Basquin[1] a considerable variation in q makes a comparatively small variation in the value of S for a given value of N.

Values of constants for the probability factor $1+kN^m$ must be very largely a matter of judgment. Referring again to the series of tests shown in Fig. 7 the minimum values of S

[1] "The Exponential Law of Endurance Tests," *Proceedings*, Am. Soc. Test. Mats., Vol. X, p. 625 (1910).

follow an exponential relation (shown by the straight line). This relation corresponds to a value of $1+kN^m$ of unity for all values of N. As noted previously, for values of N above one million, such a result seems unusual though it is sometimes found. As our test data for long-time endurance tests are few, for structural parts and machine members, whose failure would endanger life, it would seem advisable to use a probability factor of unity for all values of N in which case the proposed formula for repeated stress becomes:

$$S=\frac{B}{(1-Q)N^{\frac{1}{8}}} \quad \ldots\ldots\ldots\ldots\ldots\ldots (4)$$

$$\text{or } \log S=\log B-\log(1-Q)-\tfrac{1}{8}\log N \quad \ldots\ldots\ldots\ldots\ldots (4a)$$

For cases in which failure would not endanger life, the writers propose as tentative values, 0.015 for k and $\frac{1}{8}$ for m. These values were chosen after a study of the scanty data of long endurance tests, and represent values of S rather lower than those given in some of the long-time tests studied. The formula for repeated stress for cases in which failure would not endanger life then becomes:

$$S=\frac{B}{(1-Q)N^{\frac{1}{8}}}(1+0.015N^{\frac{1}{8}}) \quad \ldots\ldots\ldots\ldots\ldots (5)$$

$$\text{or } \log S=\log B-\log(1-Q)-\tfrac{1}{8}\log N+\log(1+0.015N^{\frac{1}{8}}) \quad \ldots (5a)$$

The determination of the value of $1+0.015\ N^{\frac{1}{8}}$ is rather cumbersome, and in Table II are given values of this factor for various values of N. Charts for the graphical solution of equations (4) and (5) are given in Figs. 8 and 9. Fig. 8 is for the solution of equation (4), and Fig. 9 for the solution of equation (5). The method of using either Fig. 8 or Fig. 9 is as follows:

Enter the diagram at the bottom at the given value of Q for the problem, pass vertically to the desired value of N (this will in general lie between two curves, and its location must be judged by interpolation). From this point pass horizontally to an intersection with the diagonal corresponding to the value of B for the material, then from this intersection vertically to the upper edge of the diagram, where the value of S may be read off. It should be remembered that S is the breaking stress.

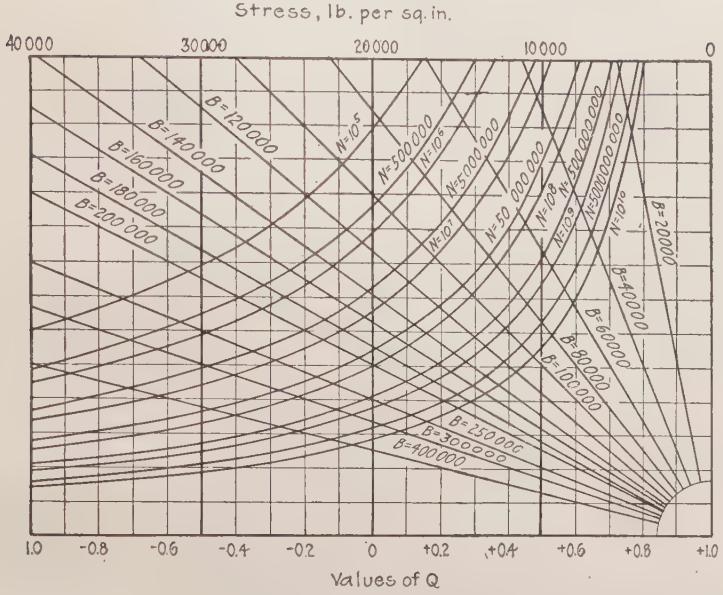

FIG. 8.—Chart for Solution of Equation 4, $S = \frac{B}{(1-Q)N^{\frac{1}{8}}}$.

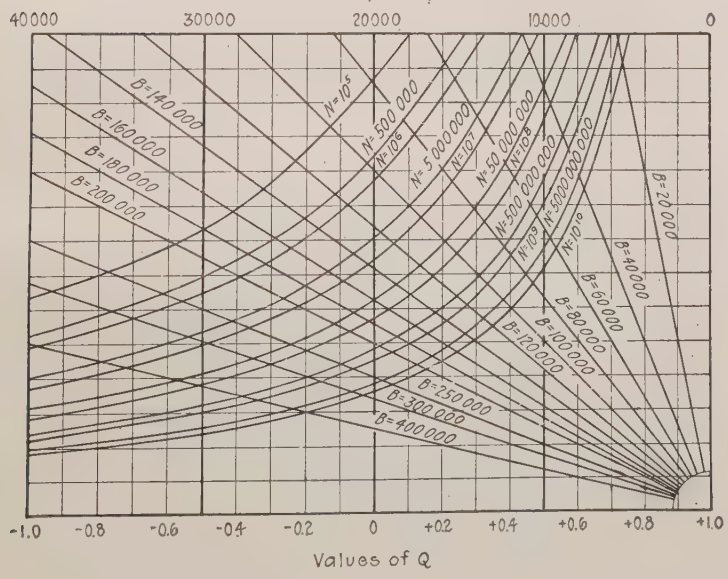

FIG. 9.—Chart for Solution of Equation 5, $S = \frac{B}{(1-Q)N^{\frac{1}{8}}}(1+0.015N^{\frac{1}{8}})$.

Factor of Safety for Repeated Stress.—In design involving static conditions the working stress must be less than the ultimate strength of the material. Reduction of stress by means of the so-called factor of safety is the only means through which a safe design is assured. It has already been pointed out that in no case must the law of fatigue be considered to hold for stresses greater than the yield point of the material, and that the requirements of static design must always be met.

When the endurance strength of a machine member is less than the yield point of the material, a factor of safety must be applied to it in order to obtain a safe working repeated stress.

TABLE II.—VALUES OF THE PROBABILITY FACTOR $(1+0.015\ N^{\frac{1}{2}})$ FOR VARIOUS VALUES OF N.

N	$(1+0.015\ N^{\frac{1}{2}})$	N	$(1+0.015\ N^{\frac{1}{2}})$
100 000	1.063	100 000 000	1.150
500 000	1.077	500 000 000	1.183
1 000 000	1.084	1 000 000 000	1.200
5 000 000	1.103	5 000 000 000	1.244
10 000 000	1.112	10 000 000 000	1.267
50 000 000	1.138	50 000 000 000	1.326

NOTE.—For structures and machines whose failure would endanger life, it is suggested that the value of the probability factor be taken as unity for all values of N. For other structures or machine parts, the values as given by the above expression and here tabulated are suggested.

Fatigue involves two factors, stress and the number of repetitions, and the factor of safety may be applied either to the stress or to the number of repetitions. Since the stress must also satisfy the requirements for proper static design, the "life" of the material or machine would seem to be properly insured by applying the factor of safety to the number of repetitions; that is, a design should be made with a stress corresponding to failure for a number of repetitions the machine is to withstand multiplied by x, the factor of safety. This places emphasis upon endurance rather than on strength, but since the stress is low when determined by the condition of fatigue, this emphasis seems properly placed. While this method reduces the stress

considerably less than would the application of the same factor of safety to the stress, it should be remembered that the function of the factor of safety is, in part, to guard against excessive stress due to non-homogeneous material, initial stresses, and local stresses due to fabrication, and that such stresses are reduced in effect after a few repetitions. Moreover, excessive stresses may be resisted for a considerable length of time without producing more than a small percentage of the total damage required for rupture.

On the other hand, it should be remembered that repeated stresses tend to destroy the primitive ductility of the material. Material has failed in practice under extremely low repeated stresses, suggesting that defective material, local flaws, and the like may play a much greater part in determining the endurance of a material than they do in determining its static strength.

Two examples of the use of the proposed formulas are given:

1. The eyebars and chords of a railroad bridge truss are to be made of structural steel, and should be designed to withstand 2,000,000 repetitions of stress, varying from dead load to dead load plus live load. The dead load is about one quarter of the live load. What value of working stress should be used?

The value of Q for this case is $+0.20$, the value of B (taken from Table II) is 110,000, and the value of N may be taken as six times 2,000,000 or 12,000,000. As the failure of the bridge would endanger life, equations (4) or (4a) are to be used. Substituting the above values in equations (4) or (4a), there is obtained for the proper value of $S = 17{,}900$, lb. per sq. in. The same result may be obtained from the use of Fig. 8. As the safe *static* stress would hardly be taken higher than 16,000 lb. per sq. in., it is seen that static conditions govern the design.

2. A line shaft is to be designed to withstand 500,000,000 complete reversals of bending stress and is to be made of a special steel for which B has been experimentally determined as 300,000. What fiber stress should be allowed? The yield-point strength of the material is 90,000 lb. per sq. in.

As the failure of a line shaft does not, in general, involve danger to life, use equations (5) or (5a) or Fig. 9. $Q = -1.0$, B is given as 300,000: Take N as six times the designed endur-

ance or as three billion. Substituting the above in equation (5*a*) or using Fig. 9 there is obtained for the working stress $S = 12{,}100$ lb. per sq. in. As this is far below the allowable static stress endurance conditions would govern in this case.

VARIOUS EFFECTS.

Effect of Rapidity of Repetition of Stress.—A certain amount of time is required for any member of a machine or structure to assume the deformation corresponding to any given load, and if repetitions of load follow each other at intervals shorter than this time, the deformation in the member, the stress set up, and the number of repetitions it will withstand may be appreciably affected. A few recent British tests[1] of material under repeated stress seem to indicate that for small members there is no appreciable effect produced by varying the rapidity of repetition of stress below about 2000 repetitions per minute. Above that speed very little test data are available.

Effect of Rest on Resistance to Repeated Stress.—If metal is stressed beyond the yield point so that plastic action is set up, its strength and its elastic action are improved under subsequent stress, if the material is allowed to rest. Recent experiments by British investigators[2] seem to indicate that, for steel and iron at least, the effect of rest on the resistance to repeated stress is negligible for unit stresses below the yield point of the material.

Effect of Sudden Change of Outline of Member.—Every sharp corner in a piece subjected to repeated stress facilitates the formation of micro-flaws in the piece. From results of repeated stress tests made by Stanton and Bairstow, at the British National Physical Laboratory, on test specimens of different shape, the superiority of the test specimens in which sharp corners are avoided is obvious. The relative values for strength

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[1] Stanton and Bairstow, "On the Resistance of Iron and Steel to Reversals of Stress," *Proceedings*, Inst. Civil Engrs. (British), Vol. 166, p. 78 (1906); also *Engineering* (London), Vol. LXXIX, p. 201 (1905). Turner, L. P., "The Strength of Steel in Compound Stress and Endurance under Repetitions of Stress," *Engineering* (London), July 28, August 11 and 25, Sept. 8, 1911.

[2] Eden, Rose and Cunningham, "The Endurance of Metals," *Proceedings*, Inst. Mech. Engrs. (British), Parts 3 and 4, p. 389 (1911).

under repeated stress for the shapes tested seems to be about as follows:[1]

Rounded fillet	100
Standard screw thread	70
Sharp corner	50

Service Expected from Various Machine and Structural Parts.—We do not know with certainty whether any material can resist an infinite number of repetitions of any stress however small. The safest view for an engineer to take seems to be that under repeated stress materials of construction have a limited "life." The exponential formula for repeated stress gives results in accordance with this view. If this view is held, the number of repetitions which any structural or machine member will have to withstand in normal service becomes of importance. The following list gives the numbers of repetitions of stress which may be expected to be applied to various machine and structural members. The list is intended to be suggestive rather than to serve as an exact guide.

The members of a railway bridge carrying 100 trains per day for a period of 50 years would sustain about 1,826,000 repetitions of stress. The stress would vary from the dead-load stress to a live-load stress averaging somewhat below that caused by the passage of the heaviest locomotives.

A railroad rail over which 250,000,000 tons of traffic passes would sustain something like 500,000 repetitions of locomotive wheel loads, the stress being slightly more severe than a repetition from zero to a maximum. The rail would have to stand, in addition to the locomotive wheel loads, something like 15,000,000 repetitions of stress caused by car wheel loads. The stresses set up by car wheel loads would be about half as great as the stresses set up by the locomotive wheel loads.

A mine hoisting rope bent over three sheave wheels and operating a hoist 100 times a day, in a term of service of five years would sustain 550,000 repetitions of stress. If the sheave wheels are so placed that they reverse the direction of the

[1] Experiments are lacking to show whether these ratios hold for cold-worked material, or for brittle material. It would seem possible that cold-worked or brittle material might be more injured by sudden change of section than ductile material.

bending of the rope, the range of stress would be nearly a complete reversal; if bending takes place in one direction only the range of stress is from nearly zero to a maximum.

The piston rod and the connecting rod of a steam engine running at 300 r.p.m. for 10 hours per day, 300 days per year for 10 years, sustains 540,000,000 repetitions of stress, and the range of stress involves almost complete reversal.

A band saw in hard service for two months sustains about 10,000,000 repetitions of stress varying from nearly zero to a maximum.

A line shaft running at 250 r.p.m. for 10 hours a day, 300 days per year, sustains during a service of 20 years 900,000,000 repetitions of bending stress due to force transmitted by belts, gears, and driving chains. The stress is almost completely reversed. It should be noted that for the line shaft the torsional stress is not repeated nearly so often as is the bending stress.

The shaft of a steam turbine running at 3,000 r.p.m. for 24 hours per day, 365 days in a year during 10 years service sustains 15,768,000,000 reversals of bending stress caused by the weight of rotating parts and the tangential force of the inrushing steam.

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DISCUSSION.

Mr. M. H. Wickhorst.—We have in rails an example of the action of repeated stresses in causing failure. Take one type of failure, the "split head," which consists of an interior longitudinal crack in the head lengthwise of the rail; this has been found to be due to the side-spread at the top of the head. We may start with a material that is structurally sound, that has no interior opening, but by the "ironing" action of the wheels and the continual addition of side-spread at the top, with material on the inside of the rail that will not stand stretching side-ways, a small crack starts which keeps on growing with repeated action and finally develops a split. This type of failure occurs mostly in segregated and spongy material from the upper part of the ingot. Much has been and is being done to improve rail manufacture, so that we may hope in time to practically eliminate the split-head type of rail failure. That is, perhaps, not the type of failure contemplated in Mr. Moore's paper, but we have another type of failure that is causing us a good deal of concern, and that has not yet been worked out, that is, the "internal transverse fissure," which seems to be particularly a type which needs to be worked out along the line of studying the action of repeated stresses. If we take a rail and bend it in the testing machine, there is a load which is sufficient to cause rupture on a single application. A somewhat lighter load, but sufficient to produce a permanent set, will finally cause rupture, if applied a sufficient number of times back and forth. If we decrease the load further, we come to a point where, with one application, the material returns to its original form, and we say it is within the elastic limit under a single application. But with a sufficient number of applications of the load, rupture can be produced, and that is where we want further light as to the limiting stress that can be safely developed under a load applied 1,000,000, 10,000,000, or perhaps a 100,000,000 times. Moreover, we want that information for what may be called normal material, as well as material that is not perfectly uniform throughout. We are beginning to suspect that rolled material such as rails

Mr. Wickhorst.

Mr. Wickhorst. may develop small internal tears in rolling. We should like to know, therefore, if there is originally a slight defect, of possibly a hundredth, several hundredths, or a quarter of an inch, what stress that material can be safely subjected to under a great many applications. If we look still further into the future, it is not impossible that we may wish to subject rails to this sort of test, not merely for investigative purposes, but perhaps also for specification purposes.

Mr. Young. MR. C. D. YOUNG.—This is a most important subject, and I am glad to see that we have been getting papers during the last two years bearing directly upon the subject. I think we can all recall that Mr. Hunnings[1] gave us some very valuable information on this subject last year, indicating that the number of repetitions of stress before rupture was dependent somewhat on the elastic limit of the material. I am inclined to believe that Fig. 5 in Mr. Moore's paper confirms indirectly Mr. Hunnings' conclusions. I am afraid, however, that this figure may be misleading in so far as the grades of the material are concerned, that is, as to the grades which may be selected advantageously for service conditions involving repeated stresses, especially where these stresses are rather high. For example, at first thought, and without the information that Mr. Hunnings has given us, the diagram would seem to indicate that for certain purposes, cold-drawn screw steel or open-hearth rail steel, probably of high carbon, would be better than staybolt iron for staybolt material. This, I believe, is not a fact. If we were to attempt to use high-carbon, high-strength material in fireboxes for staybolt iron, in view of the information here presented, I am afraid that the results would be disastrous and that detailed fractures in the staybolts would develop rather rapidly. As between iron and high-carbon steel, I am inclined to think, as has been pointed out by Mr. Hunnings, that the reason for the lower number of repetitions before rupture, with the former material, is due to the fact that the material was stressed initially above the elastic limit, while the latter was stressed initially below the elastic limit. I should like to ask Mr. Moore if that is not the case.

[1] "A New Vibratory Testing Machine and Results Obtained by its Use," *Proceedings*, Am. Soc. Test. Mats., Vol. XIV, Part II, p. 548 (1914).

I should also like to ask Mr. Moore if he has any information to indicate the value of his formula in so far as the effect of wear upon the parts under repeated stress is concerned. It is the general practice of railroad engineers to design parts for a given fiber stress well within the elastic limit. A limit of wear is then established, as for instance, for car axles, crank pins, and piston rods, and it is the wear to this limit that causes the part to be removed from service rather than the number of repeated stresses; that is, when the part reaches the minimum size, it must be removed. I think it would be of great value to know the effect of the wear upon the ability of the material to resist repeated applications of load so long as the load is well within the elastic limit, as is the ordinary practice in design. Mr. Young.

MR. H. F. MOORE.—Taking up the question raised by Mr. Young as to the relative value of cold-drawn steel and wrought iron for use in staybolts: a threaded staybolt subjected to bending develops very high local stresses at the root of the threads. Theoretically, at the root of a sharp V-thread, any bending causes a fiber stress of infinity; practically, the local stress is very high, probably well above the yield point of the material. As was pointed out in the paper, the test results and the proposed formulas are only for cases in which the fiber stress developed is below the yield point of the material. The relative endurance strengths of materials subjected to repetitions of stress above the yield point of the material can not be predicted from the results of repeated stress tests below the yield point strength of the material. For staybolts, a severe vibratory test, such as was proposed by S. V. Hunnings before this Society,[1] or such a severe vibratory test as has been proposed by J. O. Arnold, would seem to be a better test for staybolt material than an endurance test with fiber stress below the elastic limit. Mr. Moore.

MR. EDGAR MARBURG.—I should like to ask Mr. Moore whether in his examination of the literature, he found any data in answer to a question which has frequently been suggested, namely, what effect the artificial exaltation of the elastic limit, through a single application of a load in excess of the normal elastic limit, has upon its subsequent behavior under repetitive loading both for stress of the same kind, and of opposite kinds. Mr. Marburg.

[1] *Loc. cit.*

Mr. Moore. MR. MOORE.—That point involves somewhat the question raised by Mr. Young. We are, at the present time, attempting to get at that problem, starting experiments at the University of Illinois. The data we have, rather meager though it is, tends to indicate that the endurance value of materials is somewhat impaired by cold work. If the number of repetitions involved is below, we will say, 10,000, or in the tens of thousands, the cold work appears to improve the quality of the material; but if the number of repetitions is very high, say in the tens of millions, so far as the evidence we have, which is rather small, indicates anything, it shows that cold-worked material is inferior to hot-worked. This is a matter on which I should not wish to speak at all dogmatically. The little data we have indicate that certain kinds of cold work, for very low stresses, injures the endurance quality of the material, but that is a tentative conclusion which we hope to prove or disprove by later experiments.

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